The Complete Technology Book on Minerals & Mineral Processing
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Mineral is defined as a naturally occurring solid chemical substance formed through biogeochemical processes, having characteristic chemical composition, highly ordered atomic structure, and specific physical properties. By comparison, a rock is an aggregate of minerals and/or mineraloids and does not have a specific chemical composition. Mineral resources of India are sufficiently rich and varied to provide the country with strong industrial base. The country is particularly rich in metallic minerals of the ferrous group such as iron ores, manganese etc. It has the world largest reserves in mica and bauxite. In the field of extractive metallurgy, mineral processing, also known as mineral dressing or ore dressing, is the process of separating commercially valuable minerals from their ores. Mining is the extraction of valuable minerals or other geological materials from the earth, from an ore body; the term also includes the removal of soil. Materials recovered by mining include base metals, precious metals, iron, uranium, limestone, etc. There are three methods of mining; conventional or manual mining, semi mechanised mining and mechanised mining. Geopolymerisation is the processes which can transfer large scale alumina silicate wastes into value added geopolymeric products with sound mechanical strength and high acid, fire and bacterial resistance. One of many useful applications of geopolymerisation is the immobilization of heavy metals and radioactive elements. The production of non ferrous metals from natural mineral ores is, in general, highly energy intensive. Some of the non ferrous mineral sources are bauxite, granite, magnesite, limonite etc. Limestone is a sedimentary rock composed largely of the minerals calcite and aragonite, which are different crystal forms of calcium carbonate (CaCO3). Limestone processing includes several steps; primary crushing (jaw crusher, gyratory crusher, impact breaker), secondary crushing (cone crusher), fine grinding and pulverization, conveying, screening, washing, heavy media separation, optical mineral sorters, drying and storage. The non metallic mineral mining and quarrying industry segment covers a wide range of mineral extraction. Most of these minerals are found in abundance close to the surface, so underground mining is uncommon in this industry segment. Mineral resources of India are sufficiently rich and varied to provide the country with strong industrial base. The country is particularly rich in metallic minerals of the ferrous group such as iron ores, manganese etc. It has the world largest reserves in mica and bauxite.

This book basically deals with methods of mining, mining machineries, geopolymerisation of mineral products and waste, industrial and scientific aspects of non ferrous metals production, processing of alumina rich Indian iron ore slimes, limestone processing, limestone exploration and extraction, the mineralogy of asbestos, the use of asbestos and asbestos free substitutes in buildings, flotation column ;a novel technique in mineral processing, applications of thermal plasma in the synthesis of covalent carbides, nitrogenous fertilizers, manufacture of ammonium bicarbonate etc.

This book is designed to describe the details of mining and processing of different minerals like alumina rich iron ore slimes, conversion of waste to a high valued product, lime stone, asbestos, coal beneficiation, gravity concentration processes to recover values from coal and ore fines and many more. The book is meant for everyone who wants to study about the subject or wants to venture into the field of mineral processing.

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MINING

General Consideration

Production of blocks of large sizes and weight is a special feature of granite mining. As such the method of quarrying for granite as well as equipments used for the purpose differ considerably from those applied in normal mining of other ore deposits. Any means which cause fragmentation and irregular shape of mined out usually done for the sake of higher recovery will be miserable.

Granite mining is essentially by open cast quarrying. Efforts are made to select a deposit showing uniform features concerning hardness, colour shades and textures preferably having well spaced vertical and horizontal joints. These favourable features make mining of granite simpler and easier. In few countries it is customary to mine big masses and then split them into sizeable blocks as per size requirement of the buyers.

Normally deposits with little or no over burden are preferred. In case over burden covers a potential deposit it has first to be removed by drilling 30 to 60 cm deep holes of 5 cm diameter followed by blasting. Boulders which can yield marketable blocks, if found in over burden, are transported and dumped separately for sizing and dressing. The other boulders are removed and thrown in the waste dump. The exposed granite is carefully examined for its suitability to produce large blocks. Weathered portion is drilled to a depth of 30 to 60 cms, blasted and removed to dump yard. The in situ rocks mass found suitable is marked for splitting into blocks. If joints are present holes are drilled into joint to a suitable depth, usually controlled by horizontal joints and charged to an optimum level and blasted. In case no joints are present, series of holes are drilled along a chosen line of splitting at interval of 15 to 20 cms upto 30 cms depth. Feathers and wedges are inserted into the holes and carefully hammered in a calculated sequence so that the block splits along the line.

The horizontal joint - planes help in dislodging the block at its base. The splitting of blocks is generally done by taking advantage of the rift and grain. The block is then removed to dressing yard where it is dressed to required size and shape.

In respect of mining of the black granite, a greater precision and careful examination of the rock has to be made. In course of dressing of the block sometime careful examination reveals that the block has hair cracks and other defects like presence of white lines, etc. As such these blocks are rejected leading to poor recovery of desired sizeable blocks from total mined volume.

The opening of the quarry is done by cutting first a trench across its face towards the inner body. Another trench is cut at right angle to the first one and thus two long free faces are developed as indicated in plate No. IV. The first range of blocks is to be recovered from these two trenches. After this preliminary operation, the blocks are mined by drilling regular holes along chosen lines as stated above and splitting of them along these lines. The purpose of this drilling system is to relieve the workers from hard toil, ensuring the best straight alignment of the range of holes, that splitting the blocks becomes easier with sharply reduced use of explosives and consequently lower rate of granite wastage.

Granite quarrying as stated above essentially involves removal of over burden and waste rock from quarry, drilling, splitting, lifting transportation and dressing of the blocks. These operations require certain amount of mechanization at some stage or the other. Thus heavy machines like compressor with jack hammer for drilling, cranes for tilting, lifting and loading the heavy blocks, shovel for loading of waste dump, dumpers/dump trucks for transportation, diamond wire saw for recovery of well shaped blocks from in situ rock body are required for good output from the mine. Failure to invest adequately in equipments often leads many mines owners to poor returns or even losses.
Mining Machineries

The nature and quantity of machinery required for mining of granite blocks will depend on the production capacity of the mine, determined while planning and preparing the project report. For example, to have a limited number of blocks for meeting the domestic requirement of a small processing plant (generally a tile manufacturing plant) a compressor with jack hammer drills having arrangement for line drilling coupled with few sets of pegs and feathers, chisels and crowbars will be sufficient. For the purpose of lifting and loading of heavier block, provision may be made for a chain pulley block having 10 tonne capacity. On the other hand, for a medium to large size quarry producing sizeable blocks mainly for export, various kinds of equipments will be required to be used at various stages of mining operation.

The major stages involved in granite quarrying and the machinery used for the same are as follows

**Development of mining faces**
This is done by using shovel and bulldozers, which remove the over burden or weathered rocks covering the deposit. For preparing the two long free faces at right angle to each other flame jet burner is used to make two vertical slots some 10 cms wide.

**Splitting of blocks**
Compressor (12 cu. mt/min. air delivery) and jack hammers with arrangement for line drilling are required to make holes at the interval of 15 to 20 cms along chosen lines. Pegs and feathers are inserted in these holes and hammered in a calculated sequence for splitting of the blocks. After cracks are developed, hydraulic jacks are used to push the bigger blocks away from their mother rocks mass, whereas a dragging winch is used to pull the blocks away from mining faces. To prevent air pollution, dust collectors are used at the time of drilling the holes. Recently diamond wire saws have been developed to cut the blocks from insitu rock mass.

**Handling of blocks**
Chain pulley blocks (10 to 15 ton capacity) or more commonly fixed or mobile cranes (20 to 30 ton capacity), are used for tilting, lifting and loading of the blocks. These cranes also remove the rejected blocks or mine wastes from quarry faces. To effect this operation rejects are loaded on a wire mat having strong loops on four corners which are put into the dragging hook of the crane attached with the boom for throwing the quarry waste away from mining faces.

**Squaring or dressing of blocks**
After the blocks are detached from their mother rocks, they are shifted from quarry faces to dressing yards. Commonly dressing is done to have well shaped blocks free from protrusions and depressions by making use of pitcher hammers (Fig. 5), and chisels of different sizes. In well equipped mines squarring of the blocks is done by using squaring plants to have perfect flat walls of the blocks.

**Transportation**
To transport the blocks from mine site to processing plant, lorries and trucks are utilized. For export, the blocks are put into the containers placed on the lorries and are exported along with containers by shipping.

**Methods of Mining**

1. Conventional or Manual Method

Linear vertical drilling along chosen lines and wedge splitting by manual method is the most common procedure of granite quarrying. Rocks having desired quality are selected from among the rock exposures. Overburden, if any, is removed and fresh rock are exposed and checked for its suitability. Drilling and channelling are done by hand using chisels and hammers. Blocks as well as slabs of desired sizes are then cut from developed faces. Vertical joints if well spaced, rifts and grains. If any, are utilized for splitting the blocks. Horizontal joints, which are called sheetings, are used for dislodging the blocks at their base.

In course of quarrying the grey or pink granite the rock is at times heated by burning wood, charcoal or dry grass along a particular line. The heat generated splits the rock along that line and makes workers job easier. To avoid development of cracks in unwanted directions generally very little blasting is done. To dislodge the bigger blocks, mild charging of linear holes with gun powder and controlled blasting using
The recovery of black granite blocks from a quarry varies from 10 to 15% whereas those of grey or pink granite from 30-40%. Recovery depends mostly on the nature of the deposit and technique of mining.

2. Semi mechanized Method
The manual method of granite mining is labour intensive resulting into high production cost. The recovery of material in desired sizes may be enhanced with aided equipments like air compressor and jack hammers and high capacity derick cranes. For transportation of quarry wastes away from mine, a couple of trucks or dumpers may be provided in which loading and unloading can be done by manual means. The same trucks or dumpers can be utilized for carrying the finished blocks from mine to processing plant. The blocks dislodged from the parent rock can be manually dressed to desired shape and sizes. Provision of a quarry bar will facilitate rock cutting in a straight line and at right angle avoiding a lot of wastage obtained by drilling with manually held jackhammers.

3. Mechanized Method
As stated on the foregoing chapters, for a granite quarry having capacity production of 1500 and 2000 m³ per annum, mechanization will be required at various stages of activities like face development, drilling and wedge splitting, handling, squaring or dressing of sizeable blocks etc. Involvements and utilization of various kinds of equipments for mechanized mining of dimensional blocks of granite have already been described in the Para. In some of the countries complete range of machineries for quarrying and handling of granite blocks are manufactured and supplied to quarries for accelerated production of desired blocks. An illustration of such equipments and procedures with the help of which well selected and well shaped dimension blocks of export quality are extracted is attached as Plate I.

After overburden lying on the deposit is removed with the help of proclaim, dozer or pay loader, two long free faces are made. These faces are vertical and situated at right angle to each other. Thus if the deposit occurs above ground level, more than one mining benches can be prepared. Preparation of such mining benches will involve drilling, blasting and throwing away of superfluous materials part of which may also render smaller size granite blocks. The vertical faces having flat walls can be developed either by jet flame channeling (Mch. 3) or by drilling series of holes along selected lines at the interval of 15 or 20 cms with the help of pneumatic drills (Mch 3) followed by splitting of the peripheral blocks with the help of pegs and feathers (Mch. 2) and also by controlled blasting with gun powder. After the faces are ready series of holes are drilled along chosen lines and bigger blocks are detached by wedge hammering in a calculated sequence. After the cracks are formed, hydraulic jacks are used to push such blocks on a kind of cushion made of smaller fragments of quarry wastes. The bigger blocks are then dragged away from mining face with the help of dragging winch and further split up into sizeable blocks of the size specifications quoted by the buyers. Such blocks are then dressed by skilled cutters or more commonly by bush hammering and flaming machines and shifted by cranes to stock yards for final checking and dispatch.

According to the latest technique of granite mining, modern methods like jet channeling, flame cutting and various combinations of diamond saws, etc., are being used in few countries for their quarries with high production capacity. Through these latest techniques of mining, recovery of the desired granite blocks is remarkably higher, the wastage of mother rock being considerably low. On the other hand, well shaped blocks are produced in a shorter time to ensure better margin of profit.

Mining of other decorative Stones
Other stones, which are extensively used for construction and decorative purpose, are marbles, sandstones, slates, calcisilicates, limestones, etc. of various shades and colours. Most of them are flaggy in
nature and are quarried mostly by manual means. Except marbles, which are generally massive and compact, the mining techniques for all other flaggy stones are almost similar. In respect of marbles the mining methods adopted are similar to those used for granite.

A case history related to mining of Kotastone of Rajasthan highlights the quarrying procedures applicable to all other flaggy deposits.

A cushion bed with 1 to 1.5 m thickness is left between over burden and productive (splitable) zone to avoid any damage to the layers due to deep hole blasting. This cushion bed is broken manually and handled manually. Where it is too compact, it is shattered by small charge of gun powder.

Mining of productive bed is totally manual and stones of desired sizes are extracted with the help of chisels, hammers and crobars. Slabs are split up along cleavage planes and are dressed into regular dimensions. Channels/recess to a depth of 8 cms are cut manually to provide edges from where the layers can be splitted.

The wastes recovered in course of mining are loaded into trucks or dumpers and thrown into the dump yards. The useful products are stacked as per size and thickness into stock yard or directly sent to Rly, siding or polishing units for further dressing, polishing or edge cutting. The recovery from production zone varies from 40 50%.

The annual production from 55 mining leases stands at about 10 million m3 engaging about 20,000 labourers in mining, polishing, transportation and dispatch of Kotastone, Rajasthan.

Before opening a mining face the layer chart is prepared precisely to decide upon the recovery ratio and thereby the profitability.

Conservation and Safety

Like other minerals, decorative stones are also a non renewable commodity. Good black granites in particularly have very limited reserve. As such the following steps should be taken to conserve them for future use

- Adoption of modern method of mining permitting higher recovery and lower wastage of the material.
- Utilization of smaller blocks for tile manufacture. In many of the South Indian States smaller blocks are thrown away as mine wastes.
- Use of even second grade material to meet the domestic requirements.
- Taking up R & D work indigenously to evolve a solution, which can treat the hair cracks on account of which blocks are rejected and thrown away as mine waste. In Italy cementing materials are used to join the cracks.

There is a general practice to work for only such deposits, which are occurring above ground. This permits higher profitability for the mine owner because working below ground and below water table involves higher cost of production. Rigid implementation of suitable legislative measures for working below ground, will go a long way to conserve this valuable scarce natural resource.

The provisions of the M.M.R.D. Act, the Indian mines Act and the Rules & Regulations made therein apply to the mines of granite. Proper attention has to be paid to the following

Mining benches be prepared in such a way so that it takes care of fundamental safety requirements.

The use of wedges for splitting the blocks may injure the worker due to jumping in course of hammering. As such the wedges should be tied to each other with a wire or linear holes after insertion of wedges be filled up with sand or drill dust to avoid jumping of the wedges.

The workers driving the wedges into the holes should never stand on the blocks being split. If a large block is being reduced to smaller size blocks the former be stabilized before a worker mounts on it.

While driving the wedges, neighbouring workers should maintain a safe distance to avoid injury by flying rock splinters, goggles with shatter proof glass and a safety shield between adjacent workers be provided
to minimize the danger.
No worker should be allowed to push away the separate block from mother rock by foot.
While blasting the rock with light gun powder all safety rules should be strictly followed.
The cranes and all other machineries deployed at quarry must be operated in accordance with the relevant
safety requirements. For safe handling of the large blocks the crane should never be loaded in excess of its
rated capacity or its stability limit.

PROCESSING

GENERAL CONSIDERATION

The raw granite or other decorative stones attain their importance and beauty only after processing.
Carving and processing of stones in India and abroad are in practice since the dawn of civilization. Some of
the most spectacular monuments of architecture have been oriented with stones. The great wall of China,
pyramids and leaning Tower of Pisa, statue of Gomteshwar, Vidhan Sabha Bhawan Bangalore and stone
chariot at Humpy, Karnataka, Vivekanand rock Memorial Kanya Kumari, etc. are the significant examples.
Our ancestors have developed their unique style and pattern of chiseling, carving and polishing to give new
life to lifeless stones. However, the ancient processing of stones was crude and slow.
With the advent of sophisticated machineries for sawing, polishing and edge cutting enabling processing of
granite slabs of large sizes and mirror finish, the stone industries have attained a new height in recent past
creating a boom in the international as well as domestic market.
The blocks recovered from a mine are sawn into various sizes, shapes and thicknesses to suit the
requirement of the users. With the help of diamond saw it is now possible to cut thinner slabs and tiles with
mirror finish for the purpose of wall cladding and such others uses.
Processing of granites broadly involves four phases of activities, viz. (i) final dressing of the block, (ii)
sawing or slicing, (iii) polishing and finally (iv) edge cutting and polishing of edges. These activities were in
the past carried out mostly by manual methods. But now mechanical methods are more prevalent.

Manual Methods

In the early years cutting and dressing of granite block used to be mostly manual or sometimes with the
help of wire saws. Grinding and polishing was also manual, which used to be done by workers specially,
trained for the purpose. The process used to be very slow and time consuming. Subsequently the process
was replaced by crude form of polishing machineries operated manually or partly manually. The abrasives
used were of primitive types.
The crude block or slab received from the quarry site is dressed with chisels and hammers. Such blocks
are generally 2 to 5 cms oversize of the net volume for which prices are paid to quarry owners. The material
lost in course of finely dressing to get almost a perfect hexahedral block is well compensated for by higher
value. Polishing is done either for any of the six faces or all the faces according to the requirements of a
consumer. In the former case only the face to be polished is first dressed and subjected to polishing. In the
latter case, all the faces are finely dressed and subjected to polishing. After flat surface is obtained by
rough chiseling, the face is made more even and uniform by fine chiseling which is a concentration of
minutes equispaced and dense depressions almost of equal size caused by light strokes from a chisel
point. The face is now ready for grinding. The worker takes 10 to 12 hours to dress one square foot area.
Manual polishing is done by rubbing the stone with rough surfaced iron plates with polishing media like
steel shots, carborundum powder and tin oxide. Fragmented steel shots are employed in initial stages
followed by carborundum powder of diminishing grit sizes starting from 60 mesh. At the end tin oxide is
used to get a mirror like finish. Polishing of about 9 sq. ft. granite surface requires about 14 hours to obtain
glazed surface. Now this method of manual polishing is obsolete. Only sides of slabs are some where
polished manually.
Engraving of pictures and inscribing letters on polished surface is done even now by skilled craftsman by using pneumatic cutting tools or by applying sand blasting techniques. (Sand blasting film - cutting out the required pattern on the film and then exposing the pattern to a sand blast.)

Mechanical Methods
Through this method the entire processing inclusive of sawing, polishing, edge cutting and chamfering is done by machines of different designs and capacities. Even dressing or squaring of the granite block before it is subjected to sawing is done by block dressing machine, which increases sawing capacity upto 40% and reduces the cost of slicing irregular materials over the undressed surfaces of the block. The Process flow sheet for cutting and polishing of granite is given below.

1. Sawing
Sawing or slicing is a process by which granite blocks are reduced to monument, slabs and tiles of varying thicknesses suiting the requirement of the buyers. In size it varies from a small tile slicing plant to a giant monument or slabs sawing plant. The various kinds of sawing machines are described as under

*Wire saw* This has a continuous length of wire running over pulleys which is used as a cutting tool. Silicon carbide or steel shots mixed with water are used as cutting media. Wire saws being although cheaper are not capable of yielding a high rate of production. An advantage to wire sawing is that it produces a smooth cut which minimizes the subsequent dressing cost. However, its disadvantages are low rate of production, increasing cost of loose abrasives and frequent changing of wire.

Diamond wire saw is improved version of the usual wire saw machine. In this case the loop of wire is mounted with diamond bound steel beads to provide accelerated cutting action. The guide pulleys provide proper wire contact length with the drive wheel and reduce the vibration of the wire while cutting the stone. The Italian Diamantfil manufactured by Pellegrini which is a fixed plant for squaring and recutting the blocks of granite and other similar type of stones with diamond wire, has the same device of wire sawing. The rigidity of structure together with the down feed control system and the absence of vibration allows the machine to produce surfaces of the maximum precision and planarity. Once cutting has been started the machine can work without an operator since the down feed control is automatic while a series of safety devices stop the machine in the case of insufficient water supply, a break in the wire or the completion of the cut.

*Gang saws* There are various types of gang saws with oscillating motion for slicing the granite blocks in India and abroad. The gang saws of Carl Meyer, West Germany being used by TAMIN (Tamil Nadu Mineral Corporation) and MML (Mysore Mineral Limited) can take 30 to 60 blades. Steel grit circulated with a slurry of lime water acts as cutting media. The rate of down feed is 4 cms/hr and power requirement is met with by a 10 HP motor. A few of the Italian gang saws have provision for 100 steel blades which requires main motor of 30 HP and auxiliary motor of 8.75 HP. Another one with 80 blades is operated by a 25 HP main motor. The main advantage of gang saw is higher production and the fact that it can saw larger granite blocks to produce large monuments and slabs to fetch higher value. Further, to saw the slabs of 20 mm thickness, steel shot gang saw is found to be most economical.

*Circular saws* Circular saws are by far the most commonly used equipments for sawing of granite or marble blocks. Based on the depth of cutting, circular saws with variable diameter are in use.

A typical circular sawing machine consists of a stable heavy machine gantry which moves vertically on two robust machine columns. On the gantry guide, the circular saw is fixed vertically so as to slide from one end to the other. The machine is fitted with computerized control panel which helps automatic monitoring of the various stages of sawing. Depending on the hardness of the rock, required thickness of the slabs/tiles and the size of the block, various parameters of sawing like speed of horizontal movement of the saw, the RPM
of the circular blade and the down feed etc. are to be adjusted. All these parameters can be fed to the system in advance through the control panel for automatic control and execution of sawing. Lifting of the blade and positioning of the block for the next cut is also achieved by programming through the control panel. Water of required pressure is used as coolant and is supplied to the machine with a pump. The granite blocks are placed on the block carriage cemented firmly to the carriage trolley with plaster of Paris moved on rails underneath the blade and positioned suitably. For enhanced life of the blade, peripheral RPM of the blade and the rate of down feed are to be controlled properly. Higher rate of cutting may be had at the cost of usual life of the blade. Circular block saw are mainly used for slab cutting. Depending on the nature of the rock the rate of cutting varies from 0.9 to 1.5 sq meter per hour. Yet another type of machine manufactured indigenously by Shah Granite, Bombay, saws the blocks horizontally with half of the block sawn from one side and another half from the other side. The machine has a total power requirement of 110 HP. Each saw has a diameter of 1.35 m. Since the horizontal slicing machine requires much smaller saws with thinner diamond segments, the tool cost and material wastage is substantially lower than the vertical block saw required to cut the same size granite block. Further, since sawing is effected from two opposite sides simultaneously, production rate is also higher. However, the main drawback with such kind of machine is the step caused by the deviation of the saw. However, as explained by the manufacturer of this type of machine, the deviation of the saws during sawing of a hard stone like granite is inevitable, irrespective of the fact whether slicing is done by horizontal saw, vertical block saw or steel shot gang saw. The basic reason for the deviation of the blade is the tremendous normal load (bucking load) on the saw, which tends to bend the saw. In case of a horizontal slicing machine this deviation results in a slight stop in the center, which can be removed easily. In case of a vertical block saw too the deviation of the blade does occur. However, instead of leaving a step in the center, it results in a taper or in other words non uniform thickness of the slab. In the case of a steel shot gang saw, the blade also deviates resulting in unequal thickness as well as an adulating surface. As such deviation during the sawing of hard granites cannot be completely eliminated but at best it can be reduced to an acceptable level by using a good sturdy machine, good tools and efficient operation of machine.

One of the Automatic block sawing machine (fixed and portable type) manufactured by Fickert & Winterling, West Germany, has the following description.
The machine consists of the bridge on which the saw carriage runs with rise and fall rest. The bridge of the fixed type is mounted on two lateral foundation walls. The machine is equipped with a portable block carriage. The bridge of the portable type runs on rails.

**Bridge**
The sturdy steel bridge is designed in such a way in order to reduce self induced vibrations. The inverted V guides are covered with hardened stainless steel strips. These strips are wear resistant and easily exchangeable.
The bridge of the portable type is movable by sturdy cast iron travelling devices on precision rollers. The bridge runs on planed beams being equally covered with stainless steel strips. The displacement and exact guidance of the bridge is effected by rack and pinion drive on both sides. A brake prevents the bridge from being shifted during the cut.

**Saw carriage**
The saw carriage is made of cast iron and runs in the inverted V guides of the bridge on hardened, adjustable precision rollers. The feed of the saw carriage is effected by a special lead screw, driven by an infinitely variable D.C. motor with speed reducer.

**Rise and fall rest**
The rise and fall rest runs in amply dimensioned dove tail ways with hydraulic control and self adjustment. Strong and hydraulically released disc springs warrant a connection between the saw carriage and the rise
and fall rest absolutely free from play. This kind of vertical adjustment is sturdy and insensitive. Its advantage in comparison with the vertical adjusting mechanism by lead screw and nut consists in the very poor wear and allows both a very sensitive and a rapid adjustment. Moreover, the rise and fall rest is equipped with a scale and easily adjustable stop dogs for the switches limiting the upper and lower position of the saw blade.

**Work spindle**
The work spindle runs in precision ball and roller bearings. It is driven by a three phase A.C. motor with speed reducer. The gears of the speed reducer are ground or shaved and run in oil bath.

A variable speed drive unit would have the disadvantage that an over dimensioned motor with an excessive current demand would be required for the low speed of the sawing spindle.

**Operation and control**
The complete electrical control units including the control buttons for the machine, is incorporated into the control desk beside one of the machine foundations.

In order to allow the operator to be close to the block when setting, an additional pendant panel is provided.

**Block carriage**
The machine may be equipped with a very sturdy block carriage, made of steel. With its four sturdy rollers running in roller bearings, the carriage runs on rails.

With the fixed type, the lateral displacement of the block carriage within the machine can also be effected by self braking motor driven gear and rack and pinion on both sides. For the exact positioning of the block carriage, the automatic displacement is effected by quick and fine feed approach. The block carriage is clamped in its respective working position.

**Automatic sequence control of cuts**
The machine may be equipped with an automatic sequence control of cuts. The required width of each plate, even of different size may be pre selected. After each cut, the saw blade moves upwards out of the block, and the block carriage is displaced by the pre selected width.

After the last cut, the machine is disconnected automatically.

**Safety devices**
- Overload protection of the main motor by under voltage relay.
- Overload protection of the saw blade by contact ammeter. When the saw blade is overloaded, e.g. by a faulty set, rate of cutting depth or rate of feed, the rise and fall rest moves upwards until the overload will disappear.
- Protection against undue idle run by contact ammeter. In case of idle run of the machine of about 10 minutes, due to a trouble of the automatic control, the machine is stopped.
- Protection against falling off in speed (jamming protection). When the speed falls off below ca. 50% of the lower spindle speed, the machine is stopped.
- Protection against the interruption of the water supply by a control pressure gauge which stops the machine as soon as the water pressure falls below the admissible lowest value.

2. Polishing
The original colour and pattern of a natural stone become apparent only after polishing. The sawn surface of stone is polished manually or mechanically in stages by making use of various grinding and polishing media. Steel shots/grits or rough carborundum powder is used for rough polishing, which is followed, by medium and fine carborundum powder or silicon carbide. Emery powders are used in the next stage and finally mirror finish is obtained by using tin oxide. The last stage of operation is also known as buffing. A series of polishing heads arranged in a straight line over the rigid beds make up the nonstop polishing machine. Polishing discs made of mild steel copper or lead are common. Segments or bricks, of abrasive materials are often used in sophisticated polishing machines.
The consumption of polishing media and time involvement will depend on the area to be polished as well as extent of required finish. Of course, operational techniques and precision observed during the same together with quality of polishing media selected for different degree of finish in different types of stone also play important role.

The types of polishing machines, which are commonly in use, are single head polishing machine, line polishing machine and hand polishing machine.

**Single Head Polishing Machine**
The machine generally used for polishing of monuments consists of a cross bar which moves horizontally on two paralleled toothed rails on side walls. The cross bar carries the spindle which could slide perpendicular to and simultaneously with the horizontal movement of the cross bar. Depending on the stages of grinding or polishing different types of grinding or polishing heads are fixed to the head of the spindle. All the movements of the cross bars and the spindle are pre adjusted with the help of electric motors provided for the purpose. The system can work in automatic or semi automatic mode. Electric switches and sensors monitor control various activities of the system. Water is used as coolant to minimize the wear and tear of the polishing head as well as to achieve the even polishing of the surface. Generally the length of track is about 5 m and effective polishing width is about 2 m depending on the nature of the rock, rate of grinding and polishing varies from 1.5 to 1.75 sq m per hour.

**Line Polishing Machine**
This machine is used for slab polishing in big processing plant. This consists of 6 to 8, sometimes 10 spindles arranged in a line. Spindles are mounted in pairs on separate cross bars and slide along the cross bars in opposite direction from side to side. Unlike the single head polishing machine the cross bars containing spindles do not move.

The slabs to be polished are placed on a continuous conveyor belt, which moves below the row of the spindles with approximate rate of 0.1 to 0.5 m per minute. The stages like rough grinding, smooth grinding, rough polishing, fine polishing, buffing, etc. are covered from one end to other by using polishing heads containing different range of grinding and polishing media. Every pair of spindles is controlled by a separate control board and the entire process, i.e. feeding the slabs to final polishing is fully automatic. Roller conveyers are provided at input and output ends to facilitate proper loading and unloading of slabs. The grinding width ranges from 1.6 m to 1.8 m. The thickness of slabs to be processed ranges from minimum 20 mm to maximum 250 mm. The production capacity is about 20 sq m per hour.

**Hand polishing machine**
In this machine the polishing arm having the spindle carriage and the grinding wheel attached to it is moved manually over the surface to be polished. The spindle head rotates by means of electric power. This machine is generally used for polishing odd size blocks, which cannot be polished in the single head automatic machines. In hand polishing machine, grinding head can be raised to required height. Thus the blocks of any height, which cannot be accommodated in single head polishing machine, can easily be polished by hand polishing machine. Blocks having length up to 5.5 meters and width of 2.5 to 3 meters can be polished with this machine. It is a versatile machine, which can do a lot of work, which is expensive to do on the automatic machine. The quality of polish depends on the skill of the operator. Since the machine has only one polishing head and is operated manually the production is limited.

**Polishing discs**
The different types of polishing discs which are in use, are made of cast iron mild steel copper and lead. The diameters of such discs reduce with stages of polishing from coarse to fine. For initial stage of grinding and polishing keeping steel shots as grinding media generally cast iron or mild steel discs having diameters from 15 to 30 cms are used. In the subsequent stages discs having diameters from 12.5 to 10 cm are used. For the polishing copper discs with 7.5 to 20 cm diameter are utilized. In the final polishing stage lead discs having diameter from 7.5 to 12.5 cm and thickness from 3 mm to 6 mm are used. Out of all the polishing head the most common is satellite head, which is also known as planetary head. The second one is rocking head, which is also known as fickert type oscillating head. Both of these have their advantages and disadvantages. The planetary type grinds very rapidly and also gives well polished
surfaces. However, since it is high speed grinding head it needs frequent maintenance. On the other hand rocking head is simpler and cheaper to maintain. Most of the stone processors prefer rocking head. There is yet another device to grind the granite surface which is known as diamond grinding plates which grinds the surface of granite very rapidly. However, they are used when larger production is needed to justify higher fixed cost. Further the diamond grinding plates are costlier to use. Further the diamond grinding plates are costlier to use, and it is required when surface of the sawn slab is not flat.

Polishing media The quality of polishing as well as time taken for the same depends to a large extent on the abrasives being used. The abrasives for stone industry consists mostly of silicon carbide bound in a magnesite bound. The abrasives are apparently very easy to manufacture. It is for this reason that a large number of stone processors in India manufacture their own abrasives. A good abrasive will grind faster and will give better quality of polish. In Europe, there are firms specializing in the manufacture of abrasives and rarely does a granite processor make his own abrasive.

GEOPOLYMERISATION OF MINERAL PRODUCTS AND WASTE

PRINCIPLES OF GEOPOLYMERISATION

Since 1978 Joseph Davidovits has developed amorphous to semi crystalline three dimensional alumino silicate materials, which he called geopolymers (mineral polymers resulting from geochemistry). Geopolymerisation involves a chemical reaction between various alumino silicate oxides (Al3+ in IV V fold coordination) with silicates under highly alkaline conditions, yielding polymeric Si O Al O bonds, which can be presented schematically as follows

The above two reaction paths indicate that any Si Al materials might become sources of geopolymerisation. According to Davidovits geopolymeric binders are the amorphous analogues of zeolites and require similar hydrothermal synthesis conditions. Reaction times, however, are substantially shorter, which results in amorphous to semi crystalline matrices compared with the highly crystalline and regular zeolitic structures. The electron diffraction analysis conducted by Van Jaarsveld, Van Deventer and Schwartzman showed that the structure of geopolymers is amorphous to semi amorphous. The exact mechanism by which geopolymer setting and hardening occur is not fully understood.

The formation of geopolymeric materials follows much the same route as that for most zeolites, i.e. the three main steps are.

Dissolution, with the formation of mobile precursors through the complexing action of hydroxide ions

Partial orientation of mobile precursors as well as the partial internal restructuring of the alkali polysilicates and

Reprecipitation where the whole system hardens to form an inorganic polymeric structure.

As shown by Figure 1. the main difference between zeolite formation and geopolymerisation is the solid to liquid ratio in the initial reaction mixture. It is thought that for a solid to liquid ratio greater than 0.1 the mobility of species within the reaction mixture is impaired. Consequently, the Si and Al containing species described by Davidovits are unable to successfully form on ordered crystal structure (indicative of zeolite formation) before hardening of the mixture occurs.

REACTION MECHANISMS AND MATERIAL PROPERTIES

It appears that an alkali metal salt and/or hydroxide is required for dissolution of silica and alumina to proceed, as well as for the catalysis of the condensation reaction. In alumino silicate structures silicon is always 4 co ordinated, while aluminium can be 4 or 6 co ordinated. It is possible that the coordination number of aluminium in the starting materials will have an effect on its eventual bonding in the matrix. A
highly reactive intermediate gel phase is believed to form by co-polymerisation of individual alumino and silicate species. Little is known about the behaviour of this gel phase and the extent to which the nature of the starting materials and the actual concentrations in solution are affecting the formation and setting of this gel phase. A major experimental problem is that the gel phase cannot be frozen and then analysed to observe the evolution of its composition and texture. Moreover, the dissolution step that takes place under highly concentrated conditions cannot be isolated in order to study this process in the absence of a shifting equilibrium due to gel setting.

In order to enhance the understanding of dissolution mechanisms of Al-Si minerals in highly alkaline environments, Xu and Van Deventer performed ab initio Restricted Hartree Fock calculations on optimized geometries of a five-membered aluminosilicate framework rings cluster. The dissolution mechanism of the five-membered Al-Si framework rings model in highly alkaline solution has been revealed to consist of an ion pairing reaction and an interaction between the remaining broken ring cluster TOH and MOH, where T represents Al or Si and M represents Na+ or K+. The sodium cation gives a stronger ion pairing effect, which results in a higher exothermal dissolution energy when the five-membered rings structure dissolves in NaOH compared with KOH solution. As a result, the sodium atom can stabilize the remaining broken ring structure better than the potassium atom. Consequently, in geopolymerisation NaOH solution is expected to give a higher extent of dissolution of Al-Si minerals with a five-membered ring structure and to supply a better initialization as well as faster gel formation.

It is well known that geopolymers possess excellent mechanical properties, fire resistance and acid resistance. These properties make geopolymers a potential construction material, which has attracted a great deal of attention internationally in the past twenty years. Geopolymers have been used in products such as bricks, high strength tools, high acid resistance moulds, ultra high efficiency filters, high temperature resistance composite materials, protective coating materials, water and fire resistance construction materials and lightweight thermal insulation materials. Although commercial applications of geopolymers are limited at present, a recent increase in research and development activity could facilitate the wider acceptance of these materials.

In previous papers many Al-Si containing source materials such as building residues, flyash, furnace slag, pozzolan and some pure Al-Si minerals and clays (kaolinite and metakaolinite) have been studied. In fact, some research results have already been applied successfully in industry to substitute traditional cement. These studies did not consider the mineralogy or paragenesis of the individual minerals. In a subsequent study, Xu and Van Deventer (2000b) determined the solubility of selected Al-Si minerals in alkaline medium and their propensity to geopolymerise. It was shown that the interrelationship between mineralogy and reactivity of individual minerals is extremely complex, and that further research is required. More than 65% of the crust of the earth consists of Al-Si materials, so that it is most useful to understand how individual Al-Si minerals geopolymerise. Such information will enhance the commercialization of this promising new technology.

It is not required to use large quantities of calcined material as reactants in geopolymers, so that geopolymer technology gives substantially reduced carbon dioxide emissions compared with conventional Portland cement. Davidovits stated that for each tonne of cement produced there is 0.55 tonne of chemically bound carbon dioxide released from calcining of limestone, together with 0.40 tonne of carbon dioxide from the combustion of fossil fuel. Davidovits estimated that in 1987 cement production contributed to 5% of world carbon dioxide emissions, but with increased construction in developing countries like China this is estimated to reach 17% in 2015. Even if some calcined metakaolinite is used in geopolymers there is still a significant environmental incentive to replace conventional cement.

Typical FTIR and MAS NMR spectra obtained for a geopolymer are presented in Figure 2 and Figure 3 respectively. The main feature of all FTIR spectra of geopolymeric materials is a central peak between
1010 cm\(^{-1}\) and 1040 cm\(^{-1}\) that is attributed to the Si-O-Si and Al-O-Si asymmetric stretching mode. A shift in the location of the peak is a result of heavy metals contained within the geopolymer. In Figure 2, this peak is at approximately 1025 cm\(^{-1}\). This central peak is a major fingerprint for the geopolymer matrix. The peak at 559 cm\(^{-1}\) originates from double ring structures formed by Al and Si tetrahedra. The peak at 467 cm\(^{-1}\) is assigned to in plane bending of Al-O and Si-O linkages found inside the basic aluminosilicate tetrahedron. It is important to note that the location of this peak may vary between 450 cm\(^{-1}\) and 480 cm\(^{-1}\). The Si MAS NMR spectrum exhibits two main shifts at -87.3 ppm and -91.9 ppm, indicating an abundance of Si atoms connected in 4 direction via oxygen linkages to 4 Al atoms or to 1 Si atom and 3 Al atoms, the so called Si(4Al) and Si(3Al) sites respectively. This spectrum is in good agreement with the classical model of a geopolymer proposed by Davidovits which theoretically contains mainly Si(4Al) sites. In the Al MAS NMR spectrum, the peak shifts of 58.5 ppm and 2.2 ppm indicate a mixture of 4 and 6 coordinated Al. It is worth noting that different alkali and heavy metal ions do not seem to influence either the Si or Al spectra. It is possible that the coordination number of aluminium in the starting materials will have an effect on its eventual bonding in the matrix. A highly reactive intermediate gel phase is believed to form by copolymerisation of individual alumino and silicate species. Little is known about the behaviour of this gel phase and the extent to which the nature of the starting materials and the actual concentrations in solution are affecting the formation and setting of this gel phase. A major experimental problem is that the gel phase cannot be frozen and then analysed to observe the evolution of its composition and texture. Recent work by Phair, van Deventer and Smith used zirconia as an inert reference to examine the effects of a non aluminosilicate source on the chemical and physical properties of a geopolymer matrix. Zirconia was chosen as a filler material because it has well defined physical characteristics (homogeneity, stability at high temperature) and chemical properties such as high purity and it is relatively insoluble in water. Furthermore, the high structural resilience of zirconia to radioactivity makes it an ideal additive to increase the capacity and efficiency of geopolymers in encapsulating radioactive wastes. It was demonstrated that the inclusion of only a small quantity of zirconia (3% by mass) imparts a substantial increase in the compressive strength of geopolymers derived from fly ash. It was postulated that the observed increase in compressive strength is due to the formation of specific zirconia associated 3 dimensional polysialate species, which reduce the mobility of sodium while maintaining charge balance and structural stability of the matrix. Lee and Van Deventer showed that inorganic salts can act as a setting accelerator which promotes the geopolymer to set faster and acquire faster gaining of strength. It can also work as a setting retardant having the opposite effect of that of an accelerator. It was found that both the cation and the anion of inorganic salts affect the setting behaviour of geopolymers. The cation may have a greater effect on the orientation process of the geopolymeric precursor species, which are negatively charged, through electrostatic attraction. By influencing the accessibility of the reacting species to each other, the cation may promote bond formation and thus give different setting behaviour than the base geopolymer. The anion, due to its bulky spatial distribution and electrostatic repulsion to the reacting species, may also affect the bond formation and setting behaviour. The electrostatic factor introduced by the addition of inorganic salt to a geopolymer may have a significant effect on the viscosity of the reaction mixture. The more viscous the mixture, the less freely the reaction precursors can move, thus giving a faster setting geopolymer. Generally, geopolymerisation takes place in an ambient environment if the reactants are pozzolanic materials. A thermal setting and hardening with temperature ranging from 30°C to 70°C is needed in most cases when the reactants are naturally occurring aluminosilicate minerals (Xu and Van Deventer, 2000b). Kaolinite, feldspar, mica and many other aluminosilicate minerals have been studied for their potential to undergo geopolymerisation with the positive results showing that natural Al Si minerals could be the largest source for a geopolymer industry.
Figures 4 to 7 depict the compressive strength of geopolymer synthesized from alkali feldspar and kaolinite in either NaOH or KOH solution. The compressive strength of 24.5MPa and 25 MPa are achieved when Na feldspar and K feldspar are respectively geopolymerised in KOH and NaOH solution. It should be noted that these strengths are for the gel and unreacted particles only and significantly higher strengths are achieved when suitable aggregates are added. With the right combination of reactants it is not difficult to obtain compressive strengths exceeding 90 MPa after 7 days. By adjusting the composition and particle size of reactants, percentage of aggregates and reaction conditions, the strength of produced geopolymers, both early and long term strength can be optimized. For instance, a geopolymer synthesized from metakaolinite in NaOH and Na2 SiO2 solutions reached a compressive strength of 48MPa and 52MPa after one hour and 24 hours ageing at 65°C, respectively. In Russia, a twenty floor building constructed from geopolymerised slag twenty years ago using the technology of Prof. Pavlo Kryvenko of Kiev, Ukraine, is another excellent example of the long term strength and extraordinary durability of this material. Prof Kryvenko has geopolymeric samples of more than 40 years old that show no sign of degradation or ageing. At present the geopolymer research group at the University of Melbourne is studying these old samples from Prof Kryvenko in an attempt to explain scientifically the excellent durability of geopolymeric matrices.

Immobilisation of heavy metals and radioactive elements
One of many useful applications of geopolymerisation is the immobilization of heavy metals and radioactive elements. Since geopolymers possess high mechanical strength, and high acid, water and fire resistance, they have excellent long term stability and a higher durability compared with traditional Portland cement and concrete. These advantages make geopolymers potentially useful for land fill as well. Table 1 lists the percentage of metals immobilized and not released under aggressive conditions for geopolymers synthesized from metakaolinite/fly ash and kaolinite/fly ash matrices. It is noted that not only can the heavy metals Cu and Pb be successfully immobilized in the metakaolinite or kaolinite/fly ash based geopolymers, by a high mechanical strength can also be achieved in the resulting geopolymers.

A laboratory and pilot scale study was conducted by Hermann et. al. to solidify various radioactive residues in Germany by geopolymerisation with an emphasis on long term stability. The treated sludges containing radioactive residues and other wastes could be characterized by the parameters listed in Table 2. The geopolymers used to solidify the sludges were synthesized from reactive clay, slag and potassium silicate. The results of solidification are summarized in Table 3. Encouragingly, the solidification of the radioactive sludges by geopolymerisation was found to be successful, and the formed geopolymers demonstrated a sufficiently high compressive strength of 20 25 MPa. Cyclic freeze thaw and wet dry tests indicated that these geopolymers are temperature and water durable materials which are suitable for landfill. Extensive freeze thaw testing by the group of Prof Pavlo Kryvenko in Kiev has also demonstrated that geopolymeric materials have excellent durability for cold climate conditions. By using geopolymer technology based on the early patents of Joseph Davidovits, Hermann et. al have now commercialized a landfill for radioactive waste material at Schlema Alberoda in Germany. Other researchers have also confirmed that geopolymers can effectively immobilized heavy metals, radioactive residues and certain organic waste, with the resulting solids revealing excellent long term stability and contaminant retention. In addition, this technology is simple to apply and requires basically the same equipment as conventional cement mortar methods. It fills the gap between concrete based solidification methods which do not satisfy the requirements of long term structural stability, and vitrification which is too expensive for most cases in which larger amounts of sludges have to be treated.

Encapsulation of organic residue
A successful example of the encapsulation of used solvent distillation residue by means of geopolymerisation was given by Talling and Osterbacka. The treated residues consisted of 50% organic
material, inorganic binders and heavy metal pigments. The geopolymer was synthesized in situ by mixing large amounts of coal fly ash with silica fume and blast furnace slag. Approximately 8,000 drums of distillation residues from spent solvent distillation were encapsulated. The whole project consisted of several steps. Firstly, a 20 cm layer pumpable geopolymer was spread over an area of about 1,000 m². Secondly, a drainage layer was placed on top of the geopolymer layer. Another 40 cm thick layer of geopolymer was applied on the top of the drainage materials. Subsequently, the drums with solvent residues were placed together with soil on top of the geopolymer layer. Then the pumpable geopolymeric paste was poured into cavities between drums to encapsulate the waste. Again another 40 cm layer of geopolymer was applied on top of the layer of drums followed by another drainage layer, and finally soil was placed on top of the completed structure. This project completed in Marttila, Finland, serves as another successful landfill example.

Stabilisation of mine tailings
Stabilisation of mine tailings is another example of the use of geopolymers in the minerals industry. Van Jaarsveld et al. Investigated the potential use of geopolymers as capping materials for Kaltails tailings in Western Australia. The tailing samples were collected from four sites at a depth of 40 cm and 120 cm, respectively. The geochemistry of the Kaltails tailings and the total water soluble content are listed in Tables 4 and 5. A series of PVC columns with dimension of 650 mm in height and 300 mm in diameter were used to cap the Kaltails tailings. Fly ash based geopolymeric pastes were tested in the columns with addition of 65 to 70% (mass percentage) of tailings. Each potential geopolymer hardpan remained in each column for seven months before they were removed. The increasing hardness was monitored with some of data listed in Table 6. The geopolymers formed from fly ash, clay additives as well as Kaltails tailings were found to demonstrate some strength, low water permeability and high stability, and could be used potentially to stabilize mine tailings.

INDUSTRIAL AND SCIENTIFIC ASPECTS OF NON FERROUS METALS PRODUCTION

INTRODUCTION

The word nonferrous metals imply all metals other than iron. This Classification becomes too wide and scientifically ill defined. Within this group there can be many subgroups and, therefore, there are many terminologies, which are in common use. Names of some such subgroups, which often overlap, are common metals, less common metals, noble metals, reactive metals, reactor or nuclear metals, alkali metals, rare metals etc. Some of these can have further classification based on properties, applications or other criteria. Table 1 shows, as an example, a classification of rare metals. Some nonferrous metals (e.g. Mn, Cr, V, Mo, Si, Ti etc.) are often produced as ferroalloys and, therefore, generally discussed in ferrous metallurgy. Metals can be classified on the basis of different scientific criteria but these are not useful to the industry concerned with utility, abundance, price, properties etc. Scientific principles, however, from a common basis, in processing of ores, extraction and refining methods. In this sense even ferrous and nonferrous metallurgy have common features. This article mainly discusses some scientific principles relevant in winning of nonferrous metals, in general, and also discusses some newer developments possible in the future with advancement of science and technology.

RESOURCES IN INDIA

Available resources from the basis of indigenous commercial activity. If resources are abundant and not strategic then they can be exported. Yet, processing for value addition is always a more favourable option. Ongoing industrial activities and R&D create a knowledge base that generate capabilities to set up plants
based on imported resources and/or to export know how.

Table 3 lists production figures for some important minerals and the share of public and private sectors. From production point of view, there are two groups, namely, the developed (Cu, Pb, Zn and Al) and the developing (Ni, Co, Ti, W, Cr, R.E.). India has a strong nuclear energy programme under which are metals produced to meet indigenous needs. For all metals production one or more of the following aspects need increasing attention: improved characterization of available resources, upgradation/beneficiation of resources by modern scientific techniques, increased productivity in plants through stricter control of operational parameters and use of up to date technology, more stringent quality parameters for products, minimization of energy consumption, stricter environmental control and better waste recycle and/or management, and finally, exploration for and exploration of newer resources.

Regarding newer resources one can make mention of multimetal sulphides available in several locations in the country, various indigenous and imported secondary resources, polymetallic ocean floor nodules etc. Trillions of tonnes of nodules are scattered across the ocean floor. These nodules whose principal constituents are Mn, Ni, Fe, Cu, Co and siliceous ocean floor silt are collectively termed as manganese nodules. They are also potential sources of Ni and Co. Table 4 shows percentages of some major constituents and compares their availability in land and ocean floor. It should be noted that the seas contain in dissolved state nearly all metals but generally metal extraction is not economical except for magnesium. To understand a scientific basis, metals can be grouped in terms of reactivity based on some thermodynamic criterion such as hydrogen electrode potential. Table 5 shows how chemical properties can be understood on this basis. It should be noted, however, that commercial activities ultimately depend on economic and technological factors.

**THE DEVELOPED METALS INDUSTRY**

In India, today, there is significant production of Zn, Cu, Pb and Al, although each sector faces stiff global competition and numerous economic uncertainties. There is now modern technology and experts examine the position periodically to monitor progress and future requirements. With about 0.6 mt/year of production, India is self sufficient in Al. However, there are short falls in Zn (about 20% against a demand of about 250,000 t/year) and Pb (about 40% against a demand of about 90,000t/year). The situation is worse for copper where annual demand of about 250,000t is met 75% by imports. The industry has latest technology but search for newer processes and intermediate technologies must continue. There is need for recycling and environmental control. Experts have identified the R&D needs as development of newer agents, application bacterial leaching, use of newer hydrometallurgical routes employing ion exchange and solvent extraction, extraction of trace, minor and precious metals, development of newer alloys, use of mathematical models in processes, R&D on ocean floor nodules, energy minimization, enhancement of productivity and recovery, simplification of flowsheets, and treatment of secondaries.

Aluminium must play an increasingly important role in the Indian industry. The vitality of the industry will only be sustained if plants become more efficient and cost competitive and more environment friendly. Areas identified for particular R&D attention have been discussed elsewhere both the Bayers process and the Hall Heroults process need further improvements. Table 6 lists the future alumina quality projections. Constant attempts to improve things are yielding better results.

Welch, in a recent review, mentions that the energy consumption has been reduced in some plants to around 13 kWh/kg with current efficiency in excess of 96%. Efforts are directed towards constructing wettable cathode surfaces which corrode less and developing non consumable anodes, better cell design and process optimization etc. Unfortunately, alternative paths to aluminium, e.g. carbothermic reduction of Al2 O3, carbochlorination, electrolysis of AlCl3 in chloride bath etc. have not yet been economically viable. The scientific aspects of thermodynamics and kinetics associated with the aluminium electrolysis. The
industry enjoys much scope for diversification, for example, there is great need of various special grade alumina alone for various applications.

**ENVIRONMENTAL ASPECTS LIFE CYCLE ASSESSMENT (LCA)**

Process ingredients and systems which are major contributors to environmental degradation are now a days identified by making use of LCA which also serves as a basis for cooperation between the technical specialists and environmentalists of three components, viz. (1) life cycle inventory analysis which considers inputs for production emissions and pollutants throughout life cycle, (2) life cycle impact analysis which can be qualitative or quantitative, and (3) life cycle improvement analysis which aims at minimizing the adverse effects. In this, quantitative evaluations are made of the resources and energy used and waste released to the environment during the entire life (cradle go grave) cycle of the product, package or process. As an example, consider production of a 50 kW electric pump, which would need about half a tonne of coal for electricity generation and half a tonne of metal. The CO2 emitted is about 2 tonnes. Assuming a life of 20 years, the pump would consume some 1000 tonne of coal emitting 400 tonne of CO2 and consume 9000 MWh of electricity. Thus, the impact of the pump on the environment in terms of CO2 emission above is 2000 times than required for its production. Various aspects of impact of the non ferrous metals industry on the environment have been discussed more in detail in the present monograph by other experts. It should be noted; however, there are remedial measures now technologically feasible. Table 7 lists some principal wastes of the aluminium industry and possible treatment methods.

**METALS FOR SECONDARY SOURCES THE ENERGY ASPECT**

Increasing quantities of scrap and now being processed for increased production of metals. In North America and Japan, more than a third of the output now comes from scrap. There is great deal of recycling in the case of other metals too for which the figures may be approximately as follows Pb, 40 Au, 40 Ni, 20 Cu, 20 ferrous, 15 stainless steel, 80 and Zn, 5. In addition to the environmental protection such as recycling leads to substantial energy saving. Table 8 shows some figures for energy requirement for production of metals from their concentrates in theory and practice.

As is seen, the process efficiencies as usually low. Although there are scientific and technological reasons for such wide gaps between theory and practice, it is possible to effect substantial energy savings by use of correct science and technology. Recovery from secondary sources plays an important role because energy consumption is often substantial lower as indicated in Table 9.

Metals and minerals industry is most energy intensive. In advanced countries, it may amount to only 10 15% of the total volume of manufactured output, but it consumes 50% or more of delivered energy, the metal sector accounting for about two third of this. The energy intensive values of some important processes are indicated in Table 10, which compares actual fuel consumption with theoretical minimum values (approximate figures). The Table also indicates potential energy saving based on todays technology. The energy requirement for a process is based and understood in terms of a parameter called Process Fuel Equivalent (PFE).

Use of PFE eliminates many misconceptions. An aluminothermic process is not necessarily attractive from energy point of view because of exothermicity because aluminium itself consumes energy in its production. Similarly, in theory use of scrap metal also is not necessarily attractive unless it is available as a zero energy source. Sponge iron produced as an alternative to scrap iron cannot necessarily lead to overall energy economy during steelmaking.

Some people often have the misconception that because of the high temperatures involved, pyrometallurgy, in general, is more energy intensive. This is often not true because many high temperature steps, such as roasting of sulphides generate exothermic heat and hot flue gases also allow waste heat recovery. Moreover, handling of large volumes of liquors, drying, evaporation and other units operations
and, particularly, electrolytes and electro refining require significant energy in hydro- and electrometallurgy. Table 11 lists some energy requirement values typical of pyro- and hydro- and electrometallurgical operations.

EXTRACTION AS A SEPARATION PROCESS

All extraction processes involve separation of a desired phase from an input source. In pyrometallurgy, this is often achieved through a slag metal reaction. At higher temperatures, there is often closer approach to equilibrium and, therefore, it is often possible to apply thermodynamics directly. We can compare an extraction step to some common methods of separation as summarized in Table 12.

In metal extraction, there can be three basic approaches (a) produce a bulk metal and then refine. (B) produce and simultaneously purify, and (c) produce a pure compound to then produce a pure metal. Common bulk metals are generally produced by (a) and (b) whereas for rare metals often adopt method (c). In pyrometallurgy, the usual steps are drying, crushing, grinding, beneficiation, agglomeration, roasting, reduction, smelting, refining etc. The usual steps in hydro and electrometallurgy are leaching, solid/liquid separation, ion exchange, solvent extraction, precipitation, electrolysis, electrefining etc. There can be special techniques such as bioprocessing, vapour transport processes, plasma processing, vacuum melting/refining, sublimation, arc/beam melting, zone refining etc. The final steps in any refining process become increasingly difficult because the purified material tends to absorb impurities from the environment readily. Thermodynamic considerations show that a metal is more stable when it is impure. Therefore, often one has to strike a balance between quantity and quality, and purification is based on carried out in stages and in a selective manner eliminating impurities in order of priority, preferably.

Understanding of thermodynamic principles and kinetic theory can be of great advantage. It explains results achieved by a great deal of trial and error. It can be used in guiding or altogether eliminating trial and error. Many extraction methods, particularly those for rare metals, have been based on theoretical and bench scale studies alone. The fundamental concept is described now. Essentially, in all extraction and refining processes, a desired metallic value is transferred from one phase to another. This is made possible by the presence of an activity or pressure gradient at metal/slag separation, solid/gas separation, solid/liquid separation, liquid/liquid separation or whatever considers some simple examples, ferroalloys are produced with relative ease because the alloying element being at lower activity in solution allows easier reduction of its oxide. Consider extension of the idea to beryll, BeO which is a very stable oxide.

The lower the Be content, lower is aBe and more teatable is the reaction to the right. It is another matter that Be is often used as Be Cu alloy just as Mn, Si, Ti, V, Ni etc. are often used more as ferroalloys a for deoxidation and or alloying rather than as pure metals.

Consider another simple example. One may want to have nickel impurities in the slag phase during copper the metallurgy so that nickel can be subsequently recovered from some treatment of slag. Many years ago, the author imported some data on activity and solubility of different oxides such as NiO, FeO, CoO, Cu2O etc. in a standard solvent (eutectic Na2O. SiO2 K2O. SiO2). At all imperatures NiO showed distinctive low solubility values. Assuming similar results in other slag system, one can say that during copper smelting and converting, nickel will rather go to the copper metal phase rather than the slag phase.

Techniques involved in rare metal extraction are different from conventional reduction, smelting and refining processes familiarly adopted for common metals. Reasons for this which are discussed elsewhere make it imperative that they be produced first in the form of a pure compound in which pure metal is extracted. Many new developments which created initial excitement have not proved commercial viability eventually. Mention may be made of the ALCOA process based on AlCl3 electrolysis (in chloride solvent) using bipolar electrodes and continuous copper smelting (WORCRA process. NORANDA Process). However, many of the ideas generated have found applications in other sectors.
THE IMPORTANCE OF USING A MULTIDISCIPLINARY APPROACH IN THE EVALUATION OF AMMONIA LEACHING BEHAVIOUR OF MULTIMINERAL SULPHIDES

INTRODUCTION
The present day challenge to hydrometallurgical research centres upon effective utilisation of raw materials that are not amenable to pyrometallurgical processing. For example, a leaching operation is ideally suited for processing of lean and complex sulphide ores and concentrates for extraction of nonferrous as well as precious metals. Selective leaching aims at dissolving only the readily soluble metal species and not the sulphide sulphur. Any detailed investigation of the partial or total dissolution of metal values from complex sulphide ores is important theoretically, as well as for application to flotation, leaching and smelting techniques.

RESEARCH PRACTICE/METHODOLOGY
Some aspects that need to be considered critically include the overall aim of leaching studies, raw material characteristics, analysis of leach solution and residues, and experimental options and limitations. These are discussed briefly with reference to multimineral sulphides.

1. Overall Aim of Leaching Studies
The main aim has to be specific with regard to the development of a leaching process for recovering a particular metal or all metal values present in the Cu Zn Pb bulk concentrate. One is interested in knowing what is the minimum recovery desired and what is the product mix required, because this information helps reduce penalties imposed on concentrates for processing in smelters. If it is assumed that the ultimate aim is to maximize the recovery of all metal values, then one should carry out leaching studies in terms of sulphur dissolution and/or undissolved sulphur value in the leach residue. It is also necessary to define the priorities when leaching various sulphide fractions, e.g. Pb Cu, Zn Cu, assuming that industrially it is still not possible to produce individual metal sulphides of a multimineral sulphide ore technoeconomically and, at best, only bulk concentrates can be produced with good recoveries.

2. Raw Materials
Sulphide minerals are not always stable compounds. Mining, comminution and storage can alter their characteristics and compositions in an uncertain manner. Many of these sulphide concentrates are moist (3-10% moisture) and have fine particle size and, therefore, are susceptible to atmospheric oxidation during storage. It is also noted that composition is likely to change with size fraction, storage time etc. This means that the raw material has to be characterized thoroughly. A convenient way to characterize the raw material would be to use techniques such as ore microscopy, thermal analysis, X ray diffraction, chemical phase analysis etc. Any process flow sheet developed would best be used for a specific input material only.

3. Analysis of Feed Material, Leach Solution and Residues
During chemical analysis of a multimineral system interference amongst different metals often introduces errors. In the absence of accurate chemical phase analysis methods, an unusual situation arises where the entire leaching data become ambiguous. Many procedures have been recommended in the literature by earlier researchers to minimise analytical errors.

4. Leaching as a process involving Parallel Reactions
If there are a number of sulphides dissolving simultaneously (congruent leaching) the kinetics of individual
reactions are likely to be influenced by several factors such as (a) the leaching of other sulphides (thermodynamic interference) and (b) the presence of reaction products of other sulphides (kinetic factors). Moreover, many important commercial sulphide minerals possess semiconducting properties.

5. Experimental Options and Limitations during Laboratory Studies
In order to minimize uncertainties, it is possible in laboratory studies to modify the existing rate control regime by suitable adjustment of the experimental conditions. However, in an industrial operation there are practical limitations and sometimes mixed control may still prevail. Thus, pulp density may be high enough to cause reagent starvation, stirring may not be sufficiently vigorous to eliminate liquid phase mass transfer resistance, and the system may be operated in a region where changes in oxygen and ammonia concentrations influence the reaction rate.

6. Analysis of Kinetic Data in Terms of Models
There has been too much emphasis in the literature on fitting of kinetic models and evaluating kinetic parameters for leaching of both individual and multiminerual sulphides. Due to the uncertainties cited earlier, fitting a model may not be of much significance. Many of the reported data have been explained in terms of interface controlled models. The basic premise is rather restrictive and may not be valid for the following reasons the polydisperse nature of the system, uncertain shape and mineralogical composition, changes in density, porosity and pore size, and variations in magnetic susceptibility, electrical conductivity, wettability etc.

7. Selection of Experimental Conditions for Oxidative Ammonia Leaching of Multimetal Sulphides
These variables need to be fixed in certain ranges, and this cannot be done arbitrarily. Prior to selecting experimental conditions or variables, it is always advisable to evaluate critically the literature and the experimental facilities available in the laboratory. Certain guidelines are too followed considering the equipment used in a laboratory, for example autoclaves for leaching of multiminerual sulphides. These aspects are now discussed.

The working volume of the unit is very important. This is to be determined from factors such as the length of the agitator shaft, type of impeller used, etc. that contribute to the effective hydrodynamics of the system under study. Another important aspect regards the temperature pressure relationship of water in a closed vessel and how the pressure increases exponentially above a certain temperature. The maximum allowable water loading in any sealed bomb or pressure vessel is given by (0.9) (bomb volume)/ volume multiplier at maximum given temperature. Such a relationship available in the literature indicates that the increase in volume of water is small for temperatures up to 200°C. However, as the temperature is raised to higher levels, the fluid expands to fill 150% of its original volume at 321°C, and to more than 3 times its original volume at the 374°C critical point. This has to be borne in mind and a pressure vessel should not be overfilled to ensure safety. Necessary precautions are to be taken to eliminate or minimize common hazards and thereby accidents. One ought to have correct outlook for process chemistry, risk and general safety, design aspects of autoclave components, operation and maintenance of autoclaves, safety set standards etc. as described elsewhere.

If more autoclaves are available for experimentation, one has to consider their type (horizontal/vertical, length/diameter ratio), volume capacity, type of agitator and impeller tip speed, heating medium (electrical/thermal oil type) etc. and their effects on the leaching data generated. It is to be noted that mass transfer coefficients depend on factors such as the geometry of the system, the viscosity and the density of the lixiviant, the diffusivity of the species and the stirring conditions.

For any reaction system, one must also consider the concept of zeroth time that concerns initiation of the particular reaction at the desired temperature. Not many researchers realize the importance of this induction period. It is possible to investigate the dissolution of partially oxidized sulphide mineral species present in the starting feed material. The information obtained during the zeroth hour has to be taken into
account while interpreting the actual leaching data. Following these guidelines and a critical review of literature, one can now arrive at the criteria for various factors that effect leaching with emphasis on selection of experimental conditions and their control for specific results. For instance, a methodology used for selection of 9 experimental variables (temperature, agitation, time, ammonia concentration, ammonium sulphate addition, pH measurements, oxygen partial pressure, pulp density and particle size) has been recently discussed. This has led to a judicious selection of experimental variables and their ranges, hereafter known as standard leaching conditions. These are as follows

- Temperature, ambient temperature (25°C) to 135°C
- Agitation, 1080 min 1
- Leaching time, 0 to 2 h
- Ammonia concentration, 60 gpl (3.34 mole/l)
- Ammonium sulphate when added, 45 gpl (0.34 mole (1)
- pH, 11.2 with liquor ammonia, (d) as above, 10.1 with (e) as above
- Oxygen partial pressure, 150 kPa
- Solid concentration, 1%, and 10% of bulk concentrate solids
- Particle size, 200+300 mesh (63+43 µm for 1% solid concentration of pure sulphide minerals and actual bulk concentrates, and, 10% solids of single sulphide concentrates and -140+500 mesh (106+22.5 µm) for studies with 10% solid concentration of actual bulk concentrates.

Under these standard conditions, the ammonia leaching of bulk concentrates with varying mineralogical composition results in high extraction rates (>90%) of copper from chalcopyrite, zinc from sphalerite, and lead from galena. An interdisciplinary approach involving various experimental techniques, such as microscopy, X ray diffraction, thermal analysis, chemical phase analysis, surface area measurements, and electrochemical measurements (galvanic interactions), in support of a leaching study on multimetal sulphides will now be highlighted.

PROCESSING OF ALUMINA RICH INDIAN IRON ORE SLIMES

INTRODUCTION

India is endowed with rich resources of iron ores in the form of hematite and magnetite ore deposits. Hematite reserves, located primarily in the states of Bihar, Orissa and Madhya Pradesh, are estimated to be 11.9 billion tonnes with an average grade around 62% Fe. Total magnetite reserves, estimated at around 5 billion tonnes with an average grade of 35 40% Fe are found in the states of Karnataka, Goa, Andhra Pradesh and Kerala. The total iron ore production in India (sixth largest producer of iron ore in the world) is currently around 73 million tonnes. Approximately 50% of this annual production is exported in the form of raw ore, ore concentrates, pellets and iron oxide powder.

Indian hematite ores are typically rich in iron but contain unusually high alumina (as high as 7%). The current practice of iron ore washing in India results in three products, namely coarse ore lumps, directly charged to blast furnace, the classifier fines, (3.5% alumina) which with or without beneficiation are fed to sintering plants and the slimes (6 10% alumina) which are currently discarded as waste.

It is a well recognized fact that in order to enhance the competitive edge of Indian iron and steel industry, and efficient alumina removal technology for Indian iron ores is absolutely essential. It is worth noting the following facts in this context

The alumina content in iron ore fines used in sinter making all over the world is less than 1%. In contrast,
iron ore fines in India assay as high as 3.0–5.5%. The sinter quality produced from such alumina rich ore fines, is thus much poorer. The adverse effect of alumina on sinter strength productivity and its reduction - degradation characteristics (RDI) are well documented and conclusively established. Whether in the form of alumina rich lumps or sinter, the blast furnace productivity is significantly affected due to the presence of alumina in the feed. High alumina slag, which is highly viscous, requires larger quantity of flux (10% MgO) and relatively larger slag volumes resulting in an increase in coke consumption and a decrease in blast furnace productivity. According to one estimate, a decrease in alumina content in the sinter from 3.1 to 2.5% will improve the RDI by at least six points, lower blast furnace coke rate by 14 kg per tonne of hot metal and increase its productivity by about 30% under Indian operating conditions.

The generation of iron ore slimes in India is estimated to be 10–25% by weight of the total iron ore mined - the iron ore values are lost to the tune of 15–20 million tonnes every year. In addition, these slimes stored in massive water ponds pose enormous environmental hazard. SAIL alone has more than 40 million tonnes of slimes accumulated over the years. Considering the fact that iron ore production will rise to at least 100 million tonnes soon, finding means of safe disposal/utilization of slimes is indeed urgent.

**MOTIVATION FOR THE BENEFICIATION OF INDIAN IRON ORES**

Iron ores are being beneficiated all around the world including at Kudremukh in India. Several techniques such as jigs, multi gravity separator, low and high intensity magnetic separator, conventional as well as column flotation, selective dispersion - flocculation are all part of current industrial practice. Recent advances include packed flotation column, packed column jigs and centrifugal concentrators like Falcon concentrator, Kelsey jigs, Knelson concentrator for the beneficiation of iron ore slimes. Processing of hematitic ores in India at present, however, does not involve any beneficiation except for whatever rejection of silica (and to some extent alumina) occurs during washing and classification of crushed ores. Several commendable efforts have been made by Tata Steel to come up with an economically viable beneficiation flowsheet for processing classifier fines.

A comprehensive technology development programme needed to delineate the appropriate beneficiation/utilization strategy for Indian iron ore deposits must include (1) the nature of occurrence, association and liberation characteristics of the alumina containing minerals (2) a comparison of the separation efficiency of various unit operations for both hematite/goethite/kaolinite/gibbsite separation in terms of recovery grade plots (separation characteristics) and as a function of particle size (3) a preliminary techno economic assessment of the various technology options for a typical iron ore mine in the country. A schematic diagram showing the basic technological elements of an integrated strategy to utilize alumina rich iron ore deposits in the country is presented in Figure 1.

**BENEFICIATION STRATEGIES FOR INDIAN IRON ORE SLIMES**

Considering the present magnitude of the iron ore slimes generation annually, the quantities of slimes accumulated over the years, the fact that these slimes are available in already ground form and assaying reasonably high% Fe, it is obvious that if properly beneficiated, these slimes can be considered a national resource rather than a waste of nuisance value.

The alumina content of the slimes, if brought to less than 2% Al2O3 in the beneficiated product will (a) lead to better utilization of national resources (b) achieve higher mine output (enhanced production) with not much additional costs (c) reduce environmental hazards associated with storage and disposal of slimes and (d) result in higher blast furnace and sinter plant productivity.

A number of research groups in the country have explored the possibility of reducing alumina in iron ore slurries. A critical review of the earlier R&D investigations, as presented and discussed in greater detail in our earlier publications. Clearly indicates the need to carry out a comprehensive study targeted to establish an integrated strategy of utilization of Indian iron ore slimes. It must address the following important issues
A quantitative and definitive assessment of the extent of alumina reduction possible with state of the art beneficiation technology.

A conclusive evaluation of the state of the art agglomeration (sintering/pelletization/briquetting) which can successfully convert the beneficiated slimes into a product acceptable to blast furnace without adversely affecting its productivity.

A techno economic assessment of the various options available to utilize the residual waste product (it could be as high as 50%) containing high amount of alumina and iron oxide - for example, in the production of iron rich cements, recycling of iron rich wastes by thermal treatment including conversion into metallic iron and in the production of glass ceramic materials from iron rich waste.

A comprehensive eco friendly and safe technology (for example, semi dry disposal technology) to replace the conventional practice of storing iron ore slimes in tailings ponds.

One of the more important findings of earlier investigations is that alumina in Indian iron ore slimes occurs in the form of two distinct mineral constituents namely, gibbsite (hydrated aluminium oxides) and kaolinite (and other clay minerals in minor quantities). Even though not adequately quantified, the liberation studies also indicate that a substantial portion of alumina is present in the liberated form and hence amenable to separation by physical means.

The work conducted by Tata Steel on Noamundi iron ore slimes is by far the most comprehensive study available at present. Based on their data, Pradip compared the efficiencies of different unit operations including wet high intensity magnetic separation (WHIMS) and multi gravity separator (MGS). As illustrated in Figure 2, it was possible to produce concentrates at least on a laboratory scale, assaying less than 2% alumina at an overall yield of around 50% from a slimes feed analysing 7.8% alumina. Another noteworthy observation is that the separation achieved in MGS is remarkably close to the theoretical yield predicted based on the sink float tests done on the same sample of slimes. The availability of high capacity MGS units is currently a serious limitation. Based on the published literature on the beneficiation of Indian iron ore slimes, it is not possible to conclude the reasons why the yield is limited to 50% only. Is it because of interlocking and/or because of lack of a suitable separation device? Any future work on this topic must address this question.

Separation processes based on the surface chemical differences between iron and alumina containing minerals, for example, froth flotation and selective dispersion flocculation are also promising but not investigated adequately. The availability of selective reagents capable of achieving the desired separation efficiencies is a serious limitation. It has been addressed by us in our work on iron ore slimes. A brief summary of these investigations is presented in the following section.

SELECTIVE DISPERSION FLOCCULATION STUDIES ON IRON ORE SLIMES
Pradip and co workers have systematically investigated the possibility of achieving selective separation amongst hematite alumina kaolinite montmorillonite minerals, the mineral constituents representative of Indian iron ore slimes.

Two classes of commercially available flocculants namely starch based natural polymers and polyacrylamide (PAM) polyacrylic acid (PAA) family of synthetic polymers were extensively tested. Statistically designed experiments were conducted in order to compare the efficiencies of the two polymers namely starch and PAA for selective separation of hematite from kaolinite. As illustrated in Figure 3. Starch at pH 10 is found to be a much more selective reagent for this separation.

Pradip et al. have also established that as compared to the commonly used dispersant, sodium silicate, low molecular weight synthetic polymers such as polyethylene oxide (PEO) and polyvinyl pyrrolidone (PVP) are more selective dispersants for hematite kaolinite separation with PAA as a flocculant. It is interesting to note that this effect of various dispersants on selectivity is not observed in case starch is used as a
We also modified starch as well as polyacrylamides by incorporating more selective functional groups. Modified polyacrylamides containing iron chelating groups such as hydroxamates, (PAMX) were, for example found to be much more selective than PAA or PAM. The representative results on the separation efficiency of PAMX in hematite kaolinite separation are presented in Table 1.

The deleterious effect of the presence of montmorillonite in the system was also studied in detail. Flocculation experiments conducted on the synthetic mixtures of hematite/kaolinite/montmorillonite indicated that as little as 5% of montmorillonite when introduced in hematite kaolinite mixtures could lead to a marked deterioration in the separation efficiency due to relatively less flocculation of hematite in presence of montmorillonite. Kaolinite flocculation remained largely unaffected. This deleterious effect of montmorillonite could be partially mitigated by the use of modified flocculants containing more selective functional groups. As illustrated in Figure 5, the selectivity could be partially restored when modified starch mercaptan (MMS) was used as a flocculant instead of conventional maize starch (MS). The beneficial effect of a more selective flocculant like starch mercaptan was observed with synthetic dispersants as well. An interesting finding of our work is that starch, modified starches (MMS) and modified polyacrylamides (PAMX), which were found to be excellent flocculants for selective separation of hematite from kaolinite and even montmorillonite, turned out to be disastrous in hematite-gibbsite separation. In fact, we established that starch flocculated both alumina (gibbsite) and hematite (iron oxide) - equally well. It is well known that iron oxide and alumina have identical crystal structures. The results for hematite (iron oxide)- kaolinite and hematite - alumina separation using starch are shown in Figure 6 for illustration.

We also proposed a molecular recognition mechanism underlying starch interaction with iron oxide and alumina, which explains this observation. Various research groups including ours have reported on the lack of any worthwhile success in the beneficiation of natural iron ore slimes using commonly used dispersant flocculant combinations. Based on our systematic work on delineating the reasons for this observation, we attribute it to the remarkable similarities in the crystal structure as well as the surface chemical properties of the two constituent minerals of iron ore slimes namely hematite (iron oxide) and gibbsite (alumina).

While kaolinite separation is possible with certain modified flocculants synthesized by us, even in presence of minor quantities of montmorillonite, it is not enough to reduce alumina to the desired levels in the concentrate because gibbsite remains with hematite. The key to solving the problem of iron ore slimes thus lies in developing selective reagents (flocculants, dispersants and flotation collectors) for iron oxide - alumina breakthroughs on this front, in the authors opinion will have far reaching impact on the techno economics of processing alumina rich iron ore slimes. These reagents are not only essential for solving the problem of alumina rich iron ore slimes but those will also have a significant impact on the beneficiation of iron rich bauxite deposits in India. Processing of Indian red muds, rich in alumina and iron oxide will also become economically more attractive with the availability of reagents capable of iron oxide alumina separation. A sustained and systematic interdisciplinary effort by Indian researches in this direction is therefore highly recommended.

**CONVERSION OF A WASTE TO A HIGH VALUED PRODUCT**

**INTRODUCTION**

A large quantity of rice husk is generated as a byproduct of rice milling. At present, the rice husk is considered as an agricultural waste. Burning has been the primary means of disposal. Not only does
burning create pollution problems but the extremely fine silica ash is also toxic and thus constitutes a health hazard. Even careful incineration procedures cannot completely eliminate this airborne silica. Thus, burning with its attendant problems of air pollution and ash disposal has proven to be an unsatisfactory solution. Fortunately, rice husk contains the necessary carbon and silica, to provide a nearly ideal source material for production of silicon carbide. This article describes a process for converting a waste such as rice husk to nano crystalline SiC (a high valued product).

Silicon carbide (SiC) is bestowed with many attractive properties it is extremely hard it can withstand temperatures over 1000°C without undergoing significant changes in its properties it has high degree of thermal and chemical stability it is resistant to wear and erosion as a semiconductor it could operate at high temperatures and high frequencies as well as in radiation environments. All these properties make Sic a top candidate material for a host of traditional as well as sophisticated applications.

Commercial production of SiC by Acheson process, involves high temperatures reaction of silica and carbon (generally low sulfur petroleum coke). This methods requires long reaction time and produces large agglomerates with varying crystal structure and purity. The lack of control over structure and purity of SiC obtained by the above process restricts its use for many advanced engineering applications. Components intended for such applications are usually made by high temperatures sintering of powder compacts as SiC does not melt. To have improved sintering properties, the SiC powder is required to be sub micron particle size Obviously, SiC produced by Acheson process is not suitable for making dense sintered bodies unless it is ground to ultrafine size. Therefore, in recent years increasing attention has been given for production ultrafine SiC powders.

EXPERIMENTAL
A finer fraction of the semi ground raw rice husk was cleaned, oven dried and treated at 550°C for 2 h ambient nitrogen. The resultant product was in the far of fine powders. The inflight processing of heat treated fine powder was then carried out in a 20 kW r.f. plasma reactor. The details of the reactor assembly and the operating conditions are given elsewhere. Argon was used as both plasma generating gas and carrier gas. Thick coating was observed at the inner wall of the double walled, water cooled cylindrical quartz chamber. The powder could be easily scrapped off for detailed characterization. The plasma produced powder was then treated in air at 700°C in a tube furnace using nature convection to remove excess carbon. The removal excess silica was affected by treatment with HF.

The plasma synthesized SiC powder was doped with platinum (1%) and tested as a catalyst support material. The details are given elsewhere.

RESULTS AND DISCUSSIONS
Rice husk is composed of silica in hydrate amorphous form and cellulose. Proper heat treatment such husk results in a product that is primarily an intimately mixture of silica and carbon with very high surface area. When such a product is further heated in the 1400 1800°C range, a reaction occurs between silica and carbon resulting in the formation of SiC. The possible reaction of such process was given by Lee and Cutler as

\[
\text{SiO}_2 \text{ (amorphous)} + 3\text{C (amorphous)} \rightarrow \text{SiC} + 2\text{CO}
\]

The reaction time can be greatly reduced by increasing the temperature. The effect of Co from the reaction may be sufficiently significant to decrease the reaction rate. Thus, CO needs to be constantly flushed away. Both of the above processes can easily be attained in a thermal plasma reactor. Moreover, the very high temperature (104 K), step temperature gradients (106 km 1) and high quench rates (106 Ks 1) associated with thermal plasmas can be a unique route for the preparation of ultrafine SiC from rice husk.

DEFINITIONS AND PROPERTIES OF LIMES
NOMENCLATURE

Before delineating the physical and chemical characteristics of lime, it is appropriate at the outset to define some of the different types of lime, the first derivative, manufactured product of limestone. Many of the following definitions are repetitious and overlap, but these terms are widely employed in the industry and among its consumers.

Agricultural hydrate is a relatively coarse, unrefined form of hydrated lime that is mainly used for neutralizing soil acidity and for purposes where high purity and uniformity are unnecessary.

Air slaked lime contains various proportions of the oxides, hydroxides, and carbonates of calcium and magnesium resulting from excessive exposure of quicklime to air, which vitiates its quality. It is partially or largely decomposed quicklime that has become hydrated and carbonated.

Autoclaved lime is a special form of highly hydrated dolomitic lime, largely utilized for structural purposes, that has been hydrated under pressure in an autoclave.

Available lime represents the total free lime (CaO) content in a quicklime or hydrate and is the active constituent of a lime. It provides a means of evaluating the concentration of lime.

Building lime may be quick or hydrated lime (but usually connotes the latter), whose physical characteristics make it suitable for structural purposes.

Calcia is the chemical compound calcium oxide (CaO).

Carbide lime is a waste lime hydrate by product of the generation of acetylene from calcium carbide and may occur as a wet sludge or dry powder of widely varying degrees of purity and particle size. It is gray and possesses the pungent odour of acetylene.

Chemical lime is a quick or hydrated lime that is used for one or more of the many chemical and industrial applications. Usually it possesses relatively high chemical purity.

Dead burned dolomite is a specially sintered or double burned form of dolomitic quicklime, further stabilized by the addition of iron that is chemically inactive and is employed primarily as a refractory for lining open hearth steel furnaces.

Fat lime connotes a pure lime (quick or hydrated), distinguishing it from an impure or hydraulic lime it is also used to denote a lime hydrate that yields a plastic putty for structural purposes.

Finishing lime is a type of refined hydrated lime, milled in such a manner that it is suitable for plastering, particularly the finish coat. Putty derived from this hydrate possesses unusually high plasticity.

Fluxing lime is lump or pebble quicklime used for fluxing in steel manufacture—or the term may be applied more broadly to include fluxing of nonferrous metals and glass. It is a type of chemical lime.

Ground burnt lime refers to ground quicklime used for agricultural liming.

Hard burned lime is a quicklime that is calcined at high temperature and is generally characterized by relatively high density and moderate to low chemical reactivity.

Hydrated lime is a dry powder obtained by hydrating quicklime with enough water to satisfy its chemical affinity, forming a hydroxide due to its chemically combined water. It may be high calcium, magnesian, dolomitic or hydraulic.

Hydraulic hydrated lime is a chemically impure form of lime with hydraulic properties of varying extent that possesses appreciable amounts of silica, alumina, and usually some iron, chemically combined with much of the lime. It is employed solely for structural purposes.

Lime is a general term that connotes only a burned form of lime, usually quicklime, but may also refer to hydrate or hydraulic lime. It may be calcitic, magnesian, or dolomitic. It does not apply to limestone or any carbonate form of lime (although it is often erroneously used in this way).

Lime putty is a form of lime hydrate in a wet, plastic paste form, containing free water.

Lime slurry is a form of lime hydrate in aqueous suspension that contains considerable free water.
Lump lime is a physical shape of quicklime, derived from vertical kilns.

Magnesia is the chemical compound magnesium oxide (MgO) it is an important constituent in dolomitic and magnesian limes.

Masons lime is a hydrated lime used in mortar for masonry.

Milk of lime is a dilute lime hydrate in aqueous suspension and is the consistency of milk.

Pebble lime is a physical shape of quicklime.

Quicklime is a lime oxide formed by calcining limestone so that carbon dioxide is liberated. It may be high calcium, magnesian, or dolomitic and of varying degrees of chemical purity.

Slaked lime is a hydrated from of lime, as a dry powder, putty, or aqueous suspension.

Soft burned lime is a quicklime that is calcined at relatively low temperature. It is characterized by high porosity and chemical reactivity.

Type S hydrated lime is an ASTM designation to distinguish a structural hydrate from a normal hydrated lime, designation Type N, that possesses specified plasticity and gradation requirements. It may be dolomitic or high calcium and is more precisely milled than type N hydrates.

Unslaked lime is any form of quicklime.

Whitewash is synonymous with milk of lime, a dilute lime hydrate suspension.

**PHYSICAL PROPERTIES OF QUICKLIMES**

**COLOUR.** Generally quicklime is white of varying degrees of intensity, depending on its chemical purity. The purest types of quicklimes are the whitest. Less pure or improperly calcined types may have a slight ash gray, buff, or yellowish cast. The quicklime is invariably whiter than its derivative, limestone.

**ODOUR.** It possesses a faint but distinctive odour that is difficult to define. It is slightly earthy and pungent but not offensive.

**TEXTURE.** All quicklimes are crystalline, the crystallite conglomerates vary greatly in size and spacing in their matrices. Some appear to be amorphous, but they are microcrystalline.

**CRYSTAL STRUCTURE.** X-ray diffraction reveals that a pure calcitic oxide crystallizes in the cubic system as depicted in Fig. 1. The edges of the cube are 4.797 Å in length, with calcium atoms located midway in between.

Magnesium oxide possesses the same cubic crystal lattice as CaO, except that the MgO crystal is slightly smaller and denser, with edge lengths of 4.203 Å. This accounts for the slightly higher average density of dolomitic quicklime.

**POROSITY DENSITY.** The degree of porosity of commercial quicklime varies widely in percent of pore space from 18 to 54%, with an average value of about 35%, depending on the structure of the limestone, temperature, and severity of calcinations. Dead burned dolomite has much lower porosity of 8-12%.

**SPECIFIC GRAVITY.** The true specific gravity of pure calcium oxide is 3.34, but this presupposes zero porosity, a conditions that is impossible to achieve in manufacture. Values have been reported at 3.40, and lower, but 3.34 appears to be a generally recognized average value. Commercial limes may range as low as 3.0 pure dolomitic oxides may range as high as 3.5-3.6.

The apparent specific gravity varies similarly, from 1.6 to 2.8. Average values for commercial oxides are 2.0-2.2. Values for dolomitic quicklimes average about 3-4% more than the preceding. Dead burned has the highest value of all-an average of 3.2.

**BULK DENSITY.** The same variance pertaining to specific gravity is prevalent as well as the added variable of the different physical size and gradation of the quicklime particle. The range in values in lb/ft3 is 48 70 (769-1121 kg/m3), with an estimated average of 55 60 lb/ft3 (881 961 Kg/m3) for commercial quicklime of pebble size. Lump size is about 10% lower, and ground or pulverized is 12 15% greater than this average value. (The larger the particle and the more restricted the gradation, the lower the bulk density.)
Values for dolomitic average 3.4% greater than those for high calcium.

HARDNESS. Hard burned and sintered dolomitic quicklime lies between 3 and 4 on the Mohs scale. Ordinary quicklime is variable, but is usually between 3 and 2. The same broad divergence in hardness and strength in limestones is manifest in their derivative limes.

COEFFICIENT OF EXPANSION. The only values reported are $145 \times 10^{-7}$ between 300 and 700°C and $138 \times 10^{-7}$ between 0 and 1700°C. These data probably only represent the magnitude of this measurement; certainly a variance would exist with commercial quicklimes.

ELECTRICAL RESISTIVITY. Resistivity of $71 \times 10^8$ ohms/cm at $15°C$ (59°F), declining to 91 ohms at $1466°C$ (2671°F), has been calculated. The presence of nitrogen depresses values.

REFRACTIVE INDEX. The pure calcitic oxide is 1.83 and the value of commercial quicklime ranges between 1.70 and 1.82. A value of 1.736 for pure MgO means that dolomitic quicklime has a slightly lower value than CaO. Both types possess slight refractive properties.

LUMINESCENCE. All lime oxides are very luminescent at high temperatures in the calcining range of 900°C (1652°F) and higher, hence, origin of the term limelight.

THERMAL CONDUCTIVITY. It has been estimated at 0.0015 to 0.002 cal/cm3. sec.°C temperature difference, but this value may be undependable.

HEAT OF FUSION. It is doubtful whether this has ever been accurately measured. 28,000 cal/mole has been estimated as the approximate value.

MELTING POINT. Recognized values for CaO are 2570°C (4658°F) and for MgO are 2800°C (5072°F), with dolomitic oxides intermediate.

A eutectic mixture of about 50% CaCO3 and 50% CaO is reported to melt at 1240°C under high pressure of 30,000 mm.

A recent investigation of the system CaO - MgO, involving X-ray diffraction and optical methods, that may comprise the most authoritative data, reveals a maximum solid solution of MgO, in a CaO lattice of 17% weight and a maximum solid solution of CaO in the MgO lattice of 7.8% weight, both at temperatures of 2370°C. In both instances the extent of solid solution is higher than that reported by other investigators.

Melting point for the eutectic 67% CaO and 33% MgO is 2370°C for 100% CaO, 2625°C for 100% MgO, 2825°C. Figure 2 shows the phase equilibrium diagram calculated by these investigators.

HEAT OF COMBINATION. Same value as for heat of formation, given later.

BOILING POINT. Values for CaO are 2850°C (5162°F) and for MgO are 3600°C (6512°F), with dolomitic oxides intermediate.

ANALYTICAL TESTING OF LIMESTONE AND LIME

Methods of test and chemical and physical analysis of limestones and limes are increasingly important to the consumer as a means of evaluating the specific (type or grade of) product required for optimum performance among the myriad uses in which these products are consumed. Such analyses are considerably more involved (with more alternative methods) than most other basic chemicals and materials, owing to their wider range in quality and chemical and physical characteristics. For example, sulfur is generally more uniform in its chemical characteristics than limestone even granite, basalt, and trap rock tend to be more uniform in their physical properties than limestone. Certainly sulfuric acid, soda ash, chlorine, and most basic chemicals are more uniform than quick and hydrated limes per se in both chemical and physical properties. Therefore the consumer generally enjoys a much wider range of qualities from which to select, thus leading to many individual specifications or tolerances for the producer to meet. This explains the producers increasing reliance on quality control testing in manufacture and processing to ensure conformity with these requirements.

Many of these tests and analytical methods are controversial, and often there is disagreement between...
buyer and seller on the tests that are the most accurate, realistic, and equitable to adopt. Furthermore, there is appreciable inconsistency among similar classes of consumers on the tolerances they demand. For example, one steel company may be emphatic that 0.03% sulfur is the maximum amount permissible in quicklime that is consumed for fluxing steel yet another steel company making the same type of steel may be satisfied with 0.06% sulfur. As a result, a lime or limestone producer who desires to sell to a wide variety of consumers must necessarily develop a systematic program of quality control testing. Delivery refusals of substandard material by the consumer are most costly to him.

Probably the greatest influence toward quality standardization of lime and limestone in the United States and Canada are the specifications promulgated by the American Society of Testing Materials. Many consumers may ignore these ASTM materials specifications and methods of tests. Since adoption of these standards is strictly voluntary. Yet these specifications do comprise a guide or basis on which a consumer can mold his own individual specification by modifications. These standards are jointly prepared by the consumers. Producers, and general interest members, that presumably these standards should be objective and unbiased-equitable to all concerned. Two ASTM committees are involved Committee C 7 on Lime has prepared lime materials specifications for numerous uses. Sampling procedures and methods of test for chemical and physical analyses for both lime and limestone Committee D 4 on Road and Paving Materials has written many tests and specifications applicable to limestone and other mineral aggregates for all phases of construction in which the physical characteristics are the controlling factor. There are also federal specifications and test procedures, but these are usually almost a duplication of ASTM. Some foreign countries have similar specifications, but these are generally instigated by their governments. Some of these ASTM tests are too laborious and time consuming for quality control testing by producers and consumers except as a periodic check or when maximum accuracy is needed in event of a dispute between buyer and seller. Because of this such detailed tests are regarded as referee tests. For quality control testing often special rapid short test are contrived and are widely employed, since they provide reasonably or sufficiently accurate results for routine testing. Some of these short tests along with other supplementary investigative procedures will be reviewed briefly along with the more important ASTM standards.

Sampling. More than any other factor, the accuracy of tests on limestone aggregate is contingent on how representative the sample is. In testing graded aggregate the principal pitfall is segregation of coarse and fine fractions, obscuring their true gradation. Considerable care is necessary to minimize this inherent tendency of stone samples to segregate, and sampling procedures of ASTM D 75 for stone and other aggregate is recommended. This includes sampling of stone from ledges or quarries from outcroppings of field stone and boulders at the crushing plant after final blending and screening at point of delivery. The minimum weight of the sample depends on its maximum particle size, as shown in Table 1.

Hardness, Toughness. Tests employed to determine hardness, toughness, and resistance to abrasion and impact of all types of highway aggregate are described briefly as follows

The Los Angeles Abrasion Test (ASTM C 131) is a severe accelerated test for measuring the abrasive resistance of limestone. It involves testing different specified weights and gradations of stone in the Los Angeles machine, which comprises an enclosed, hollow steel cylinder into which an abrasive charge of a prescribed number of steel spheres (adjusted for the different stone gradation classifications) are tumbled together by a rotating mechanism. After the specified number of revolutions at 30 33 rpm, the sample is screened to determine the abrasive loss in weight of the stone and from this is calculated the percent of wear of the stone. Specifications for maximum percent of wear generally range between 30 and 60%, contingent on quality of stone available and stringency of requirement.

The Deval Test (ASTM D 289) is another accelerated abrasion resistance test. It consists of a small cast iron cylinder that is inclined at 30° with the horizontal axis, around which it is rotated at 30 rpm 5 kg of
stone, consisting of 50 cubical shaped fractions, is introduced into the machine and subjected to 10,000 revolutions. The loss in weight due to attrition is calculated by sieving through a No. 12 mesh (1.68 mm) screen, and the percentage of wear is then determined. The Deval test may also be modified into an impact test by introducing steel shot with the stone. This accentuates abrasion resulting in a higher percentage of wear.

The Page Impact Test I (ASTM D 3) measures the toughness of rock by determining the resistance to impact. In a specially designed machine a small cylinder of stone, cored from the rock to be tested, is placed on an anvil and on the stone specimen is laid a 1 kg plunger. A 2 kg hammer is then dropped on the plunger from successively increasing heights of 1 cm (0.4 in.) until the core fractures. The final height in centimeters from which the hammer falls determines the toughness of the rock. There are modifications of this hammer test.

The Dorry Hardness Test is still another accelerated abrasive resistance test. A small core of stone, specially machined for the test, is placed in firm contact against a revolving steel disc on which 30-40 mesh (0.6-0.4 mm) quartz sand is fed. After 1000 revolutions the loss in weight of the stone core is calculated. The coefficient of hardness is equal to 20 weight loss/3.

Compressive strength tests with hydraulic presses in which crushing strength of the stone in psi (KPa) is measured are rarely employed, since most stone is regarded as amply strong in compression for highway uses. Furthermore, it is a costly test, since stone blocks or cylinders must be precisely machined so that both sides are completely level and smooth.

Of the preceding tests the Los Angeles Abrasion Test is the most widely utilized. It is the most severe test but is also regarded as the most realistic in simulating the abrasive action of traffic on highways.

**Specific Gravity.** There are various calculations on specific gravity of stone that reveal the degree of density, porosity, and absorption and provide an indirect indication of the hardness and weatherability of the stone. Two types of specific gravity are determined (ASTM C 127 128)

Bulk specific gravity of the stone is defined as the ratio of weight in air of a given volume of a permeable material, including both its permeable and impermeable voids, in relation to weight of an equal volume of distilled water.

Apparent specific gravity is the ratio of weight in air of a given volume of the impermeable portion of a permeable material (the solid rock) in relation to weight in air of the same volume of distilled water. In effect this is the same as the bulk specific gravity minus the volume of permeable voids.

These values are obtained from the so called wire basket test. A 5 kg stone sample is oven dried to constant weight (A), immersed in water for 24 hr, then removed and surface dried by hand with a towel and weighed (B). The sample is then placed in a wire basket, and its weight in water (C) is measured.

A quick test for specific gravity can be measured with the Beckman Air Comparison Pycnometer. This has been employed successfully with hydrated lime and whiting.

The unit weight of a fine, coarse, or mixed aggregate is governed by ASTM C 29, which prescribes three alternative methods of weighing the aggregate in a specified size cylinder that is dependent on the maximum size of the aggregate. These three procedures that comprise rodding or jigging or shoveling techniques are also contingent on the maximum size of aggregate that is present and serve to consolidate the aggregate to the maximum extent. The net weight of the consolidated aggregate is determined.

**Size and Gradation.** The physical sizes and gradations of stone are determined by screening the material through standard round or square hole wire cloth or plate sieves (ASTM E 11 70). A method of sieving by overdrying the aggregate for plus No. 200 mesh (74 µ) sizes is stipulated in ASTM C 136. Both the percents passing or retained on the different sieve sizes are recorded.

For measuring size of mixed aggregates finer than the No. 200 sieve (74 µ) a procedure is outlined in ASTM C 117. This consists of washing a sample of the aggregate in a prescribed manner. The decanted wash...
water containing suspended and dissolved materials is passed through a No. 200 sieve. This might consist of clay, stone dust, and water soluble extraneous salts. The loss in weight from the wash treatment is calculated as percent by weight of the original sample. This is the percentage of material finer than the No. 200 sieve.

A dry method of sieving mineral fillers, most of which pass a No. 200 sieve, is described in ASTM D 546. The constancy of gradation is expressed by the fineness modulus of an aggregate. It is an empirical number determined by adding the cumulative percentages retained on, say, the Nos. 100, 50, 30, 16, 8, 4, and 0.375 in., 0.75 in., 1.5 in., and 3 in. (0.15 76.2 mm) square opening sieves and dividing this sum by 100. This provides a figure of the average size of a given gradation. However, different gradations may possess approximately the same fineness modulus. This factor may be specified to control the uniformity of graded aggregate for Portland cement concrete, and specifications may require that the variation in the fineness modulus not exceed 0.2.

Standard aggregate gradations for various construction uses.

**Soundness.** Associated with the degree of absorption of a rock is soundness, since generally the more porous the stone, the more moisture it will absorb, which accentuates its susceptibility to weathering. If the moisture freezes, a disruptive, expansive pressure is exerted by the ice lenses that induce the aggregate to spall, defoliate, or disintegrate. Aggregates vary greatly in their resistance to the deleterious effect of freezing and thawing cycles. Also visible predictability of an aggregate’s soundness is speculative, since the size, shape, and strength of the stones pore structure, which cannot be observed, influence the severity of disruption through frost action. Therefore, accelerated laboratory tests have been contrived to evaluate soundness and weatherability. The most widely employed soundness tests are the sodium sulfate or magnesium sulfate tests (ASTM C 88).

This consists of immersing the aggregate samples in a saturated solution of either sodium or magnesium sulfate at 70°F (21°C) for 18 hr. The sample is then removed, oven dried at 215 220°F (101 104°C), and then cooled to 70°F (21°C). This cycle of immersion and hot drying is repeated usually five times (or a specified number of cycles) and the percent of loss is determined. Usually the limiting percent is 10 20. For stone used in trickling filters of sewage plants an extremely severe requirement of 20 cycles is often specified with only 10% loss. Only relatively few sources of limestone can meet such a requirement. Use of Mg(SO₄)₂ is more disruptive than Na₂SO₄. This test is regarded as unreasonably severe by many engineers in spite of widespread application, and other special freeze thaw tests have been introduced. However, no test yet advanced appears to be generally acceptable and realistically indicative.

The presence of an excess of alkali (Na₂O and K₂O) in aggregate may create a deleterious effect on Portland cement concrete, particularly if the cement or mixing water is also high in such alkalis. In order to test the soundness of a combination of aggregates, cement, and mixing water, the so called mortar bar test, an alkali reactivity test with cement aggregate mixtures, is employed (ASTM C 227). This is an involved, detailed test with all conditions, equipment, and materials meticulously prescribed. Briefly, mortar bars are made from one part Portland cement to two and one fourth parts graded aggregate by weight. After molding, they are stored in a moist closet for 24 hr then they are removed from the mold. The length of the bar is measured and returned to the mold in the damp closet. Daily or monthly recordings of lineal changes (usually expansions) are made for 28 days, one year, or any specified length of time. At conclusion total expansion is calculated along with observable physical changes in the bar, such as warping, cracking, surface mottling, and so on. This alleged test resembles more of a research project very complicated, costly to conduct, and lacking in reproducibility.

Another soundness test (ASTM C 289) is a chemical method of determining the potential reactivity of aggregate with alkalis in Portland cement. It is indicated by the extent of reaction during 24 hr at 80°C
between NaOH solution and the aggregate that has been crushed and screened to pass a No. 50 sieve (0.3 mm) and retained on a No. 100 sieve (0.15 mm). This is also a complicated test, but it is more realistic and simpler than the mortar bar soundness test.

**Stripping in Bitumen-Aggregate Mixtures.** Aggregate varies in its ability to retain a bituminous film in the presence of water. The bitumen aggregate combinations that possess poor retention cause the aggregate to strip or ravel from the pavement. ASTM D 1664 describes coating and static immersion procedures to determine if stripping occurs or the degree of stripping prevalent. This test is only applicable to cut back and semisolid asphalts and tars. Much of this test is dependent on visual observation, so it is only generally and not quantitatively indicative. There are many modifications of this test as well as other stripping and bituminous durability tests, one of the most important of which is the Immersion Compression test developed by the U.S. Bureau of Public Roads.

Although the reaction of aggregates with bitumens cannot be predicted with certainty, as a generalization the acidic types of rocks are more prone to strip than basic types, like limestone and slag. Acidic stone and aggregate are classed as hydrophilic, since they possess a stronger affinity for water than asphalt the basic aggregates are called hydrophobic. Since they possess better adhesive qualities for bitumens, even under wet conditions.

**Petrography.** When the texture, colour, grain size and pattern, or geologic origin of the limestone is of importance, polished thin sections of the stone in question are subjected to microscopic examination. Such Petrographic analysis is more related to research information that testing, since no limits can be set, except as pertaining to reflectivity. Otherwise, the information obtained is utilized arbitrarily or as engineering judgment. The most sophisticated microanalysis instrument appears to be the scanning electron microscope for these studies.

Other miscellaneous tests for use as construction aggregate include:

- Determination of the percent of flat and elongated fractions in a graded aggregate, since cubical shapes of stone are most desired. This is controlled by specifying a maximum allowable percentage for odd shapes.
- Determination of percent of lightweight pieces and soft particles in coarse aggregate that would prove to be unsound.
- Determination of fire resistant qualities of aggregate.
- The extent of clay lumps in natural aggregate.
- The tendency of limestone to polish under simulated action of traffic is covered by ASTM D 3319. Several states have established arbitrary criteria to eliminate aggregate that contribute to slippery pavements in bituminous surfaced roads.

**LIMESTONE USE SPECIFICATIONS**

There are 21 ASTM use of aggregate specifications for many construction purposes. There are countless modifications of these specifications written by many diverse consumer groups, state highway departments, public works department, U.S. Engineers, U.S. Navy, Bureau of Reclamation, consulting engineers, architects, and so on. Generally one authoritative group, the American Association of State Transportation Officials (AASTO), endorses the ASTM standards.

**Reflectivity.** The degree of whiteness is an extremely important determination in limestone, chalk, marble, and shell whiting and precipitated calcium carbonate, although there is a dearth of uniformity in the many individual consumer requirements. Reflectivity measurement can be obtained with the G. E. Spectrophotometer at 550 wavelength (similar to green tristimulus filter) or equivalent equipment. It is also applicable to hydrated lime. Details on this measurement are contained in specification C 110.

**LIMESTONE CHEMICAL ANALYSES**

Analytical chemical test methods on the composition of limestone, including its impurities, are adequately
Methods that is equally applicable to both limestone and lime, such as quantitative determinations of impurities, will be reviewed later in the section under Lime Chemical Analysis. Those tests directly applicable to limestone, such as loss on ignition, CO2 content, free water, and organic content, will be described briefly as follows

**Loss on Ignition.** Limestone weighing 0.5 or 1.0 g is placed in a weighed platinum crucible, covered with a lid, and then heated gradually in an electric muffle furnace up to about 1000°C. The sample is maintained at this temperature until a constant weight is obtained. This means that all volatiles have been expelled. The difference between the original weight of the sample and the final weight represents the loss on ignition.

**Mechanical Moisture Content.** Limestone weighing 1 g is placed in a fast bottomed weighing bottle and is heated uncovered in a ventilated drying oven at 120°C for 2 hr. After heating, the bottle should be quickly sealed, cooled in a desiccator, and then unsealed just before weighing. The loss in weight represents only mechanical or hygroscopic moisture, no other volatile.

**Carbon Dioxide Content.** There are two standard methods of determining CO2 content. The first involves boiling 0.5 g of limestone with dilute HCl (11) in a small Erlenmeyer flask attached to a condenser from above. The CO2 gas evolved from this reaction then passes through a drying and decontaminating system of CaCl2 and anhydrous CuSO4 to absorption tubes filled with soda lime, which entraps the CO2. CaCl2 desiccates final traces of water. Air, free of CO2, is introduced gently throughout the system during the test and at a greater rate after the test. When CO2 is being absorbed, the absorption tubes heat up readily. When these tubes cool, they are disconnected and allowed to stand in the balance case until two weights, taken thirty minutes apart, agree within 0.5 mg. Soda lime used in this test must be porous.

The second (alternative) method is quite similar to the first except that a specially designed Midvale bulb, packed alternatively with glass wool, P2O5, and ascarite is used to absorb the CO2 that is evolved. This bulb is detached and weighed with a second Midvale bulb as counterpoise. Free moisture is desiccated and acid volatiles are recovered separately so that a true CO2 content is obtained by difference.

**Organic Content.** Determination of the organic content of limestone is extremely difficult to measure with accuracy and no simple, completely accurate method has been advanced. (ASTM makes no provision for this test.) It is usually calculated by determining the free carbon content. One method consists of liberating the CO2 by boiling the stone in acid and passing the volatiles, including the free carbon, into a mixture of sulfuric and chromic acids that oxidize the organic matter. In other tests the volatiles are boiled off and filtered through asbestos, which collects the carbon for subsequent organic combustion of the free carbon. At best most organic values are simply close quantitative estimates.

**PHYSICAL TESTS OF LIME**

There is only ASTM specification (C 110 71) embracing physical tests on burned lime products, but it is an omnibus type of specification since it includes tests on residues of quicklime and hydrated lime, standard consistency of putty, plasticity measurements, soundness of hydrate lime, popping and pitting, water retention, slaking rate, and settling rate. Other analytical methods not recognized by ASTM will be included whenever appropriate, but before describing these tests a review of sampling procedures on lime and limestone (ASTM C 50 57) is propitious.

**Sampling.** The importance of agreement between producer and consumer on where the sampling will take place—at producers plant or at destination—is emphasized. Since a producer can be victimized by the carrier in careless handling, improper protection, and delayed shipment that vitiate quality, from his standpoint sampling at his plant is preferable. Stress is placed on expeditious sampling so as to minimize exposure of the lime to air. (With limestone there is no problem in this particular.) Samples should be taken in triplicate
and immediately sealed in air tight, moisture proof containers. Each sample shall weigh at least 5 lb (2.25 kg), provided no screen size specified.

One sample should be immediately delivered to the consignee the second sample is for the consignor, if requested the third is retained with the seal unbroken for a possible independent test.

To obtain a representative sample of lump or granular material in bulk, 50 lb (22.5 kg) are sampled for each unit of 30 tons of bulk material, even though 5 lb (2.25 kg) may only be required for the test. When lime is stored in piles or carloads, care should be exercised not to select material from the top or bottom. Ten shovelfuls are recommended to be taken from different parts of the pile or carload, at least 1 ft (0.3 m) below the surface. The 5 lb samples are obtained by mixing and quartering all the shovelfuls. When sampling is effected at producers plant from conveyors or bins, a similar volume of material should be taken at regular intervals instead of all at once.

With packaged material at least 1% of the packages should be sampled with at least 1 lb (0.45 kg)/ bag being removed. Total sampling should then be mixed and quartered to provide the (3) 5 lb samples.

With powdered material in bulk, a sampling tube is specified that will remove a core at least 1 in. (2.5 cm) in diameter and is sufficiently long to penetrate into the interior—not the bottom or top. A minimum of 50 lb shall be taken from each 30 ton unit, mixed and quartered into the 5 lb triplicate samples.

At the laboratory lump or granular material to be tested should be ground to pass a No. 100 sieve (0.15 mm).

In case of rejection the manufacturer must be notified within one week after completion of tests with the cause of rejection stated. Each of the contracting parties may make claim for a retest within one week of the original test report. Expense of retesting must be borne by the party demanding it. Should the parties still be in disagreement, the third sample of material should be delivered unopened to a mutually satisfactory referee laboratory for test, and their findings are binding on both parties.

Residue. The insoluble matter (core and impurities) in quicklime is determined by weighing the residue and calculating its percent from the weight of the original sample, which is at least 5 lb. Lump material should be first crushed so that it passes a 1 in. sieve pulverized material is tested as received. The sample is then slaked carefully into a putty of maximum yield and is allowed to stand for 1 hr. The putty is then washed through a No. 20 sieve by a stream of water at moderate pressure. Washing is continued until the residue remaining visually appears to be nothing but coarse, sand like particles. The residue is then dried at 212-215°F.

With hydrated lime a 100 g sample is placed on a No.30 sieve (0.59 mm) that is nested above a No. 200 sieve (74 µ). The material is washed through the meshes by a stream of water. No material must be washed over the sides of either screen. Washing is continued until both sieves are clear of hydrate, but in no case for longer than 30 min. Residues from both screens are then dried to a constant weight in an atmosphere free of CO2 at a temperature between 212 and 248°F (100-120°C). The percentage residue on each sieve is calculated, based on the original weight of the sample, and reported separately or cumulatively.

Standard Consistency of Putty. Hydrated lime weighing 300 g is mixed with sufficient water to form a thick putty and stirred to assure intimate mixing. Type N hydrate putty is stored, covered with a wet cloth for 16-24 hr before testing. Type S hydrate putty can be tested immediately after preparation.

The equipment employed is a modified Vicat apparatus. Putty is added flush to the top of a nonabsorbent mold, 4 cm (1.6 in.) height, 7 cm at base, and 6 cm at top, which rests on a glass base. A plunger is lowered in contact to the putty surface and an initial reading is taken. The plunger is released for 30 sec and another reading is taken. Standard putty consistency is obtained when a penetration of 20 mm ± 5 mm is obtained in 30 sec. If putty is substandard in consistency, the sample is returned to original putty. More water is added, mixed for 2-3 min, and a retest is made as above. If penetration then exceeds standard, the sample should be discarded and a new one prepared.
Plasticity Determination. A special apparatus known as the Emley Plasticimeter is used in this test. A similar type of mold as employed in the standard consistency test is lubricated with water, placed on a porcelain or disposable plaster base plate, and filled flush to the top with a lime putty of standard consistency. The mold is then removed carefully and vertically without distorting the mound of putty. The base plate with putty is on a turntable. A motor rotates the turntable at one revolution in 6 min, 40 sec and rises a thirteenth of an inch per revolution. Before rotation is commenced, a disc from above is placed in contact with the paste. This is intended to simulate the action of a trowel applying lime putty to an absorbent wall base. Timing is critical. The turntable should not be started in motion until exactly 120 sec after the putty is first added to the mold. Thus, the following times must be precisely recorded:

- Starting time.
- Starting motor (and rotation).
- Record scale reading each minute until completion of test.

The test is complete when

- The scale reaches 100 or
- The scale value falls (reading less than previous one) or
- The scale reading remains constant for three consecutive readings or the specimen has visibly ruptured or broken bond with the base plate.

Essential to some degree of reproducibility in this test is a base plate with the proper degree of absorption that has been standardized at not less than 40 g of moisture absorption when the paste is immersed in water at room temperature for 24 hr. Meticulous care of base plates is necessary—cleaning thoroughly, drying, and soon-to maintain proper degree of porosity.

OTHER PLASTICITY TESTS. The Emley test is the only test recognized by ASTM. However, even adherents of it readily acknowledge its shortcomings, particularly lack of reproducibility caused principally by base plates that lack uniformity in suction as well as the varying skills of operators. Yet in spite of such criticism it still is the most satisfactory method thus far advanced and certainly is informative on the rheological properties and workability of limes. However, this method has only been adopted by the United States and Canada.

The United Kingdom employs a nonabsorbent flow table for measuring workability. The number of times the flow table is bumped is recorded in flattening a truncated cone of putty of standard consistency to three times its original diameter. On this test the British specify a minimum of 10 and 13 for hydrated lime and quicklime putty, respectively—values that would be regarded as extremely low in the United States, probably substandard.

A so-called Carson blotter test will also indicate in a crude way the degree of plasticity. A glob of putty of standard consistency is placed on a filter pad or blotter and is spread over the surface with a spatula or putty knife. The number of strokes required before the putty loses its moisture and balls up under the spatula is an approximate measure of plasticity, although obviously of completely unpredictable reproducibility.

Water Retention (and Flow Values). An indirect measure of plasticity and workability of lime in sanded mortars is the water retention test, since frequently lime that possesses a high water retentive capacity has a correspondingly high Emley plasticity value (and vice versa) with limes of low water retentive capacity. Lime weighing 500 g and standard Ottawa sand weighing 1500 g are intermixed with a measured amount of water in a nonabsorbent bowl. Hydrated lime is first wetted in the water before the sand is added or if lime putty is employed, it is first converted to a slurry before sand is gradually added, amid stirring and kneading action with a gloved hand.

The apparatus initially used for this test is a flow table mounted on a vertical shaft in such a manner that it can be raised and dropped a fixed height of 0.5 in. (12.7 mm) by means of a rotating cam. Table surface is
of nonabsorbent, non corroding metal and should be so calibrated that it slowly revolves on each drop (1
revolution for every 25 drops). A nonabsorbent flow mold, 2 in, in height, 4 in. (5 and 10 cm) in inside
diameter at the base, and 2.75 in. (70.5 mm) inside diameter at the top, is placed on the center of the flow
table and filled, flush to the top, with the above sanded lime mortar. The mortar is pressed into the mold
with fingertips to assure complete filling without voids, and the mold is carefully removed. The table is then
dropped 25 times in 15 sec. The mortar flow is the resulting increase in diameter of the flattening mortar
mass, expressed as percent of original diameter. This standardizing value is an essential prerequisite to the
water retention test itself. If the flow is less than 100%. More water is added to the mortar until on retesting
the flow measures 100 115%. The rejected mortar is returned to the original bowl and remixed for 135
seconds. If the flow exceeds 115%, the sample should be discarded and a new batch made.
The water retention apparatus consists essentially of a water aspirator controlled by a mercury column
relief and connected by way of a three way stopcock to a funnel on which rests a perforated dish made of
nonabsorbent material. A specified grade of filter paper is then fitted flatly and tightly to the bottom of the
dish and moistened.
Immediately after the flow test described previously, the remaining mortar on the flow table is returned to
the original mortar bowl and remixed for 30 sec. It is then rapidly distributed uniformly, without compaction,
over the sheet of dampened filter paper in the perforated dish, flush to the top of the dish by use of a
straightedge. The dish is returned to the funnel, and a vacuum is applied. After 60 sec of suction of water
from the mortar, the stopcock is turned off in order to expose the funnel to atmospheric pressure. The
mortar is then removed from the dish with a spatula and returned to the mold on the flow table, where it is
filled completely with gloved finger compaction flush to the top. A second flow value is then obtained in the
same manner as previously described by dropping the flow table this value is known as the flow after
suction. The whole test (or what appears to be a series of tests) should be conducted as rapidly as possible
and should never exceed 30 min from start to finish.
A high value or a percent of 85 95 indicates that a lime possesses high water retentive capacity by resisting
the artificially imposed suction, described earlier. A value of 75 85% would indicate low to intermediate
water retention. In contrast to lime, a portland cement mortar would only possess 50 60% water retention.
This test is most indicative of all mortar tests. Since water retention is an essential characteristic for use
with absorbent masonry units. Otherwise, such porous units would quickly absorb moisture from a mortar of
low water retentivity before it could harden properly and a poor bond at the mortar unit interface would
result. That is why it is an almost invariable requirement in all masonry mortar specifications.
Differences in the water retentivity capacity of mortars composed of different limes, cements, and
proportions of both can be empirically observed by a crude quick blotter test. A glob of unsanded lime putty
of standard consistency or a flow of 100 115 is placed on filter paper (150 lb/ream). The filter is immediately
inverted and the number of seconds from its application to the appearance of moisture soaking through the
paper is recorded. Contingent on the test conditions, such as percent of flow, thickness of paper, and
atmospheric humidity, 4 30 sec might expire before visible moisture occurs.
Soundness. Unsoundness in limes, characterized by cracking, pits and pops, bulging or distorted surfaces
of a freshly applied neat lime putty, gauged putty, or sanded putty, is caused primarily by presence of
coarse particles, particularly +No. 30 mesh (0.6 mm) materials, and to a lesser extent +No. 200 mesh (74 μ)
particles may be contributory. The presence of unhydrated oxides (generally MgO) in the lime is also a
source of potential unsoundness because of the possibility of delayed hydration. Actually careful milling of
the hydrate in an air separator at the point of manufacture to remove all coarse particles is the most
effective sinecure against unsoundness, and in a similar manner removal of all coarse particles from
quicklime putty by screening is likewise effective. If the gradation of a hydrated lime reveals a virtual
absence of coarse particles, then from a practical standpoint there is no need for a soundness test, except
possibly in the case of the controversial normal dolomitic hydrated lime. Yet before modern hydrate milling techniques were perfected, unsoundness from coarse particles was a chronic problem. A cumbersome, highly subjective pitting and popping test was formerly utilized to evaluate the soundness of lime hydrates, particularly for plastering. By achieving much finer milling, this test has been almost completely abandoned in the United States.

**Expansion.** The ASTM lime committee has resisted adopting a test to measure unsoundness, characterized by expansion from delayed hydration of unhydrated oxides, largely because no accelerated test yet advanced is regarded as reasonably emulative of field conditions. An autoclave test is cited in Specification C 110 that involves molding under pressure hydrated lime or lime cement limestone sand mixtures into tablets. It may be of qualitative significance but not quantitative, and it lacks reproducibility. England, South Africa, and other countries employ the Le Chatelier mold test (or slight modifications of it) in measuring potential damaging expansion in lime. These molds are filled with a workable mortar composed of varying ratios of lime, cement, and standard sand, respectively, such as 116, 219, or 3112. After being allowed to set for 48 hr, they are steamed for 3 hr and the extent of linear expansion is measured. One permissible limit of expansion is 10 mm.

**Sand Carrying Capacity.** This characteristic of mortar materials has some relationship to plasticity and workability, although it has never been correlated and it may not be directly proportional in a quantitative way. However, as a generalization, the most plastic limes possess the greatest sand carrying capacity, which means they can be mixed with larger volumetric proportions of sand and still provide adequate workability without being oversanded. Limes of poor to moderate plasticity have less sand carrying capacity and Portland cement averages 50–75% less capacity than a far, plastic lime, as determined by the Voss Extrusion Energy machine (also called Plastometer). This apparatus provides the only realistic test for measuring this property, although it is not included in the ASTM standards and rarely in any specification. The test consists of extruding a mortar under confined conditions in a cylinder and measuring the pressure exerted in the extrusion. If the mortar extrudes completely and easily and at low pressure, it is judged workable and carrying the optimum amount of sand. Compressive strength. Although pure lime is never employed in mortar for its mechanical strength, compressive strength requirements for lime and lime cement mortars are frequently specified. With hydraulic limes compressive and tensile strengths would be of some importance, much more than with fat (pure) limes.

The criteria for compressive strength tests on mortars are largely ASTM C109 75, a specification promulgated by the ASTM cement committee. It consists of molding 2 × 2 in. (5 × 5 cm) mortar cubes and then deviating from the above specification in the method of curing the cubes. Instead of underwater curing specified for cement mortars, curing is performed in a moist closet, or with cycles of wetting and drying, or in the case of straight or very high lime content mortars, just laboratory air. Cubes are then broken at 7 and 28 days with strengths reported in psi (kPa).

The setting time of fat lime mortars (without cement) is of no interest and is only applicable to hydraulic lime, cement lime, or straight cement mortars. The same standard test is employed a penetration test with the Gilmore needle and Vicat apparatus (ASTM C266 74).

**Settling Rate.** ASTM has recognized the following settling rate test for many years:

Ten g of lime hydrate is placed in a 100 ml glass stoppered, graduated cylinder and is wet down with 50 ml of CO2 free distilled water at room temperature. It is mixed thoroughly for 2 min, including inversion of the cylinder by hand. The contents are allowed to stand for 30 min and then are diluted to the 100 ml mark with more CO2 free distilled water. After further mixing, contents are allowed to stand for 24 hr. Sedimentation height at bottom of meniscus is then visually taken at 0.25, 0.5, 0.75, 1, 2, 4, and 24 hr intervals.
Slaking Rate. Probably the most equitable slaking rate test for quicklime is the current ASTM tentative standard, contained in C 110, Physical Tests for Lime, since allowances are made for different types and qualities of quicklimes. However, this and all slaking tests are invariably controversial. The procedure consists of preparing the quicklime sample—No. 6 sieve size—as rapidly as possible to prevent air slaking.

**Reactivity is defined** High reactivity is completely slaked within 10 min medium is completely reacted within 10–20 min low requires more than 20 min for complete reaction.

Readings are continued until temperature rise is less than 0.5°C (0.9°F) in three consecutive readings. Total active slaking time will be computed from the first of these three readings. This initial starting temperature is subtracted from the final temperature to obtain the total temperature rise. Before completion of slaking, temperature rise can be similarly computed for any earlier reading, like 30 sec. Values are plotted on a curve.

There are many slaking rate tests, but most are analogous to the preceding. Many lime manufacturers check lime reactivity in a very cursory manner by simply mixing approximately a like amount of quicklime and water and visually observe the reaction. There is nothing learned quantitatively from such a crude, quick test, but it can be meaningful to a person experienced with lime. Any appreciable change in slaking rate will be quickly detected.

The American Water Works Association in their AWWA Standard B202 77 for Quicklime and Hydrated Lime has contrived a different slaking rate test from ASTM. They evaluate limes by the rapidity of slaking in achieving a temperature rise of 72°F (40°C). That is, in 3 min or less it is highly reactive 3–6 min, medium reactive and over 6 min, low reactive. They do not differentiate between types of lime and specify 400 ml water to 100 g of No. 6 mesh (3.36 mm) high calcium quicklime.

Gradation. Standard mechanical sieving based on sieve sizes established in ASTM E 11 70 is virtually the only gradation test required for all lime and limestone, except whiting, which employs other methods of particle size determination.

Sieve sizes extend down to the No. 400 sieve (37 µ), but the lime industry has generally standardized on the No. 325 sieve (44 µ) as being the lowest practical sieving limit to employ for reasonable accuracy. The standard dry sieving method is satisfactory for quicklime and limestone but is totally unsatisfactory for hydrated lime. With the latter a wet sieving procedure, is strongly recommended. Otherwise, on dry sieving hydrates agglomerate, even though they adhere loosely together, and will not pass through to the small sieve sizes, thereby distorting the gradation and making it appear to be coarser than it actually is. However, in wet sieving with a stream of water applied at low pressure, the agglomerates readily break up into countless microparticles, usually of subsieve sizes, and pass through the finest screens. With hydrates most of the gradation specifications are utilized to control or limit the coarse fractions (residues), such as a maximum of 0.5% retained on the No. 30 mesh (0.6mm) and a maximum of 15% retained on the No. 200 mesh (74 µ). A few individual consumers specifications are known to require 95 or 98% passing a No. 325 mesh (44 µ), but this would represent the most stringent gradation requirement for hydrate.

For quality control testing of No. 325 mesh limestone whiting, wet sieving is also practiced, generally adhering to the test procedure 1 61 developed by the Pulverized Limestone Institute, which, after wet screening, involves drying, mechanical agitation, and final weighing of the No. 325 mesh residue. However, other evaluation tests are employed largely for research and general technical information that pertain to particle size and its distribution. These are employed more for determination of subsieve (micron sizes) the shape and form of the microparticles, whether they are cubical, plate like, or needle shaped the range in surface areas for different particles and the average surface area for a representative volume or weight petrographic analysis (thin sections) molecular structure and even the porosity (pore size distribution) of these microparticles in angstrom units. The increasing recognition that surface area is of
greater significance than particle size for many applications of limestone flour, whiting, and lime will probably lead ultimately to specifications predicated on a minimum surface area. (This method is now employed as standard practice for Portland cement in ASTM specifications C 150 76a, C 204 75, and C 115 74.) At least, one U.S. lime manufacturer uses the Blaine Air Permeability method that measures surface area in quality control testing of hydrated lime and quicklime putty. Thus, in the future lime consumers may conceivably specify that hydrated lime must possess a minimum surface area of, say, 15,000 or 20,000 cm²/g.

LIMESTONE PROCESSING

The need for individualization that has prevailed in exploration for deposits and the extraction of the stone is just as compelling in the design of a plant for conversion of the stone into marketable or usable products. The same variables exist—the physical characteristics of the stone to be crushed and screened, the chemical requirement (impurity tolerances) that demand conformance, and lastly, and at this juncture the most important requirement, the sizes or gradations of stone that are desired. Thus, a plant with adequate capacity, tailor made to the particular terrain and sizes and quality of stone required, is essential. There are no foolproof, detailed blueprints for such a plant. In fact, if an existing modern plant of highest efficiency was duplicated exactly, it might easily prove to be totally unsuitable at a second location. So in designing a new plant it is prudent to seek advice from engineering consultants specializing in this field and from manufacturers of equipment for stone processing. In view of this situation, the succeeding pages treat this subject generally with emphasis on practical plant design considerations and suggestions on avoidance and correction of operating pitfalls.

For the specifics of plant design, past issues of Pit and Quarry and Rock Products contain numerous valuable case history articles on many varied plants, providing specifications (and brand names) of equipment and performance data, and clearly demonstrate how individualized each plant is. For the countless modifications in the basic types of equipment, the most complete information source is the annual pit and Quarry Handbook and Purchasing Guide, published by Pit and Quarry in Chicago. Almost every modern type and model of equipment now in commercial use in the limestone, lime, and aggregate industries are illustrated and described in detail. Figures 1 depict flow diagrams of typical modern stone processing plants.

Obviously even though numerous ranges of stone gradations may be desired as primary products, other plus, minus, and possibly intermediate sizes are also accumulated. These undesirable sizes should be marketed as by products, the return from which enhances the profits of the primary products. This principle is equally valid whether the stone products are sold or are consumed to produce more highly refined products, like whiting, lime, and portland cement. In the event that these submarginal sizes and gradations are unmarketable or unusable, they have to be stockpiled at some convenient location on the premises, like overburden. Such material is classified by the industry as spalls (or spall piles). Again, as in the case of overburden, spall accumulations should be deposited in an area that does not interfere with plant processing and quarrying. It is bad practice to intermingle overburden and spalls. These piles should be segregated, since subsequently a profitable outlet for this stone may emerge otherwise this potential value is usually dissipated in a heterogeneous waste pile. The cost of subsequent screening and classifying is often too formidable.

Primary Crushing

Next to blasting, primary crushing is the most effective method of stone size reduction, far more satisfactory than secondary blasting. However, for high activity it is essential that the crushers size be correlated with the city of the bucket of the shovel or loader used in loading the blasted. The crusher should be able to
accommodate the largest sized stone in the bucket can efficiently handle. Proper selection minimizes the pace of oversized rock jamming the crusher and obstructing the flow of it. A large rock may pass through the crusher but then interlock edged with smaller stones, forcing the crusher to a standstill. There is absolute protection against stone jams. When jamming delays occur, they are corrected by cumbersome hand labour by prying rocks loose with the metal crowbars or using pneumatic hammer pavement breakers. A costly piece of auxiliary equipment, called a hydraulic impulse breaker, aid to be able within minutes to fracture the boulder into manageable is that clear the opening. Electrically connected and under remote control the impulse breaker is handled by the crusher operator from his control it thus reducing downtime and averting dangerous hand labour. The machine with variable speed control imparts the necessary impacts to fragrant the rock. Another crusher plant design detail that minimizes rock instructions is to install an apron, pan, or vibrating feeder that functions under a bin or hopper and induces a steady, smooth conveyance of stone to the crusher. This also tends to prevent choke feeding that generates are fines, which are undesirable in some plants.

Basically, stone is crushed in two ways by compression or impact impression, typified by the jaw and gyratory primary crushers. Consists of a slow application of load (as pressure) on rock. Since a piece of rock must be restrained to remain under load, an equal and opposite force mates shear stresses within the stone causing it to crack and rupture. In contrast, impact on which impact breakers and hammer mill crushers are indicated subjects the stone to sudden force, that is, to sharp, rapid, repeated blows. Since for limestone most primary crushing is performed by a jaw and gyratory crushers and impact breaker, only brief mention is made of other primary crushers, such as the single and double roll crusher, which are compression types. These units are mainly used for softer species of limestone and other minerals. The hammer mill, closely related in crushing action to the impact breaker, is employed to some extent but its prime particle is in secondary crushing.

Of these types the jaw is the oldest mechanical crusher and until 1960 and the gyratory dominated primary crushing of limestone. Since then, impact breakers have become of equal importance, particularly for cement and other uses that desire a high percentage of fines and small fractions. Since a number of manufacturers fabricate all these types, it is possible to obtain unbiased, reliable information on their performance for a specific situation. Many widely varying conditions affect a crushers selection, such as size of loading bucket, top stone size into crusher, desired crusher capacity, hardness and moisture content of stone, desired gradations and sizes (product mix), estimated idle time, and so on. Some large operations may use two or three types for different gradations. No single type is superior per se, only for individualized conditions.

Jaw Crusher. The original Blake or double toggle, dual jaw type is still widely used. Modifications to this are the single toggle or overhead eccentric type and another with two moveable jaws. The Blake type has one stationary jaw against which the stone is compressed by the movable jaw. The discharge end of the jaw is adjustable, providing some latitude in accommodating different top sizes. When the flow of raw stone into this crusher is controlled with a feeder, a slightly smaller crusher can be used. Then grizzlies are optionally employed for scalping off clay and stone funes.

When both jaws have smooth steel surfaces, the shape of the crushed stone tends to be more flat and elongated, similar in shape to what the gyratory yields. However, if the surface of one jaw is corrugated steel, a larger percentage of cubical shapes results. The jaw is much lower in profile than the gyratory, needing only about half as much headroom for the same capacity unit. Also wider settings can be made at the discharge end than with the gyratory. But for the same size unit the gyratory has over twice as much capacity as the jaw, and the largest capacity gyratory has five to six times greater capacity than the largest jaw. In capacity, Jaws range between 20 and 600 tons/hr. Sizes of jaw crushers range from 24 × 36 in. to 66 × 84 in. (0.61 × 0.92 and 1.67 × 2.3 m). This range of sizes crushes down to 3 5 in. (7.5 12.5 cm) top...
size, about the same as the gyatory. In balance there appears to be a consensus that the jaw is less costly than the gyatory for lower capacities.

**Gyratory Crusher.** This crushe consists of a conical head inside an outer concave bowl. Crushing is impelled by a slow gyratory or eccentric (not rotary) movement of the conical head that compresses the stone against the fixed concave crushing surface. Its crushing force remains uniform. Its capacity is greater than those of all other types of primary crushers, ranging from 225 to 3000 tons/hr. The physical size of these units ranges from 26 to 72 in. (0.66 1.9 m), with the smallest discharge openings ranging between 3.5 and 9 in. (8.8 22.5 cm). Compared to the jaw for equal capacities, the gyatory weighs and costs less. It also requires slightly less horsepower than the jaw for equivalent capacities because of lower friction. Its main disadvantages are greater headroom, more restricted discharge openings, yielding of only a small percent of cubical stone (mostly flattish) and, sometimes, greater maintenance costs. However, when high, continuous capacity is required, this unit usually provides the most economical crushing system.

**Impact Breaker.** The impact breaker consists of one or two massive impeller breakers that rotate at speeds of 250 1000 rpm and shatters rock of comparable size to the other two crushers. But this initial impact is only the start. Fragments of the original large rock are hurled at high velocity against steel breaker bars inside the crushing chamber as well as colliding with flying rock, causing further breakage and reduction in size. Pieces of stone that are still oversized go through another cycle of violent impact and collision until they are small enough to escape through the adjustable breaker bars or grate. In effect, this is a form of closed circuit crushing. Thus, this machine offers a higher ratio of size reduction than either the jaw or gyatory. But increasing speed and narrowing the openings of vertical and horizontal bar spacings, crushing is intensified, producing sizes down to 1.5 2 in. (3.75 5 cm) top size-smaller than other primary crushers except the hammer mill. Thus, if the primary product is coarse aggregate or lime kiln feed, secondary crushing may be unnecessary, except possibly for by products. Cubical shapes greatly predominate from impact crushers, more than from any other crusher. Because the breakage from ricochet and collision occurs without additional horsepower, energy requirements are less. Capacities range from 300 to 2000 tons/hr. The double impeller breaker possesses the greatest capacity. With all primary crushers when stone is still too large to convey from the crushe down chutes to a conveyor belt, it may flow directly into another feeding hopper either for the secondary crushe or onto a conveyor belt in closer proximity. This conserves belt life from the attrition of heavy falling stone. This practice is judicious when stone fines and clay are present, since fines lubricate the chute, causing the stone to slide at higher velocity. An alternative is use of a grizzly chute that permits fines to fall on the belt first, cushioning the descent of the larger stone.

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