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Gums, Adhesives & Sealants Technology (with
Formulae & their Applications) 2nd Edition



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Naturally occurring polysaccharides from plant exudates have been in use from many decades in immense quantities. Natural gums are natural polymers, which mainly consists of carbohydrates sometimes with small amounts of proteins and minerals. Gum and its derivatives are widely used in various industries as per its needs. The appearance and properties of natural gums determine their commercial value and end use. Due to their extraordinary, unrivalled technological & functional properties gum is used in many industries. Gums not only modify viscosity and consistency, they also often attenuate odour, taste and flavour intensity. Adhesive or sealant is a mixture in a liquid or semi-liquid state that is capable of holding materials together by surface attachment. Adhesives and sealants are used as a raw material for the manufacturing industry or for the service of different processing industries. Adhesives and sealants virtually touch every part of our lives. The adhesives and sealants are two chemically similar but functionally different groups of formulated products. There is no end in sight to the new materials, new formulation, and new uses to which adhesives and sealants will be put in the future.

Some of the fundamentals of the book are advantages of adhesive bonding, hybrids and coupling agents, adhesive films, designing polymers for adhesives, fundamentals of adhesion, designing polymers for adhesives, thermodynamics of adhesion, casein and mixed protein adhesives, lime-free casein adhesives, foil to paper laminating adhesives, casein and protein blend glues as wood adhesives, chemistry of protein blend glues, natural rubber adhesives, vulcanizing latex adhesives, solution adhesives from natural rubber, halogenated butyl rubber, butyl rubber and poly isobutylene lattices, polysulfide sealants and adhesives etc. This book covers a wide range of polymeric adhesives and sealants, gums along with their essential formularies, distinguished by applications and based on technology. The main areas covered in details are the basic fundamentals, properties, uses and applications, formulations and chemistry, methods of manufacturing and lastly testing methods. This book will be very resourceful to its readers who are just beginners in this field and also to upcoming entrepreneurs, engineers, existing industries, technologist, technical institution etc.

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Starch is natural polymer, available in very large quantities and at relatively low and stable prices. It consists of glucose units chemically bound together so as to form a nonreducing polyhydroxy material. Because of the many hydroxyl groups, starch has a high affinity for polar substances such as water or cellulose. Starch can be reduced to low molecular weight sugars by enzymes called amylases, or by acid hydrolysis.

Some major changes in starch usage in adhesives and related fields are the large increase of cationic and amphoteric starches in paper manufacture and the increased use of high amylose starches in the corrugating industry.

The increased emphasis on recycling makes the use of starch desirable because amylases are able to degrade the starch with essentially no effect on the major part of the adherent matrix. There is a patent on the use of amylase as a release agent in starch glued materials recycling.

Most of the starch used in adhesives in the United States is produced from corn or maize. There are four commercially available starch types that are used in adhesives. These are waxy corn starch, regular corn starch, high amylose type V corn starch, and high amylose type VII corn starch. The major difference between these starches is in the amount of amylose contained in them—approximately 0, 28, 55, and 70%, respectively, by iodine titration. Other starches usable in adhesives include sorghum starch, potato starch, tapioca starch, wheat starch, rice starch, and sago starch. The term sago was originally applied to starch from the stem of Metroxylon-type palm trees, but is often used to describe starches from other palm trees, or even applied to some varieties of tapioca starch. Some characteristics of several commercially available starches are shown in Table 1.

Regular corn starch consists of two major fractions that can be separated by precipitation with butanol under appropriate conditions. The fraction that precipitates out is called amylose. Amylose is essentially linear in form, stains blue with iodine solutions, tends to form a rigid gel from concentrated solutions or to precipitate from dilute solutions, and is about 95% digested by beta amylase (a test for strict linearity or lack of branching) when very carefully isolated. Amylose forms strong, water-resistant films when a solution is evaporated.

The other main fraction is called amylopectin. Amylopectin is highly branched (one branch every 14 to 27 glucose units), stains brown to purplish with iodine solutions, tends to remain in solution at room temperature, and is digested to about 55% by beta amylase. Amylopectin forms weak, water sensitive films. Regular corn starch (when carefully fractionated) contains an additional intermediate fraction. This fraction is precipitated out of the original corn starch solution by butanol, but is not reprecipitated when water and butanol are added to the amylose fraction dissolved in dimethylsulfoxide. The intermediate fraction is precipitated by iodine from the various solutions, and is 5-7% of the starch in regular cornstarch. The full analysis for regular cornstarch is 25-27% fractionated amylose, 68% amylopectin, and 5-7% intermediate fraction.

Table 1. Commercial starches. Approximate Data and Ranges.

-

Starch	Corn	Wheat	Rice	Tapioca	Potato	Sago
Source	Seed	Seed	Seed	Root	Root	Pith

Granule size in diameter microns	5-26	3-35	3-8	5-35	15-100	10-70
Gelatinization						
Temp.($^{\circ}$ C)	62-72	58-64	68-78	49-70	59-68	60-67
Amylose (%)	28	25	19	20	25	26
Amylose (DP)	480	-	-	1050	850	Amylopectin (DP)

When waxy corn starch is carefully fractionated, 2% intermediate fraction is found. The remainder is amylopectin.

High amylose corn starches differ from regular corn starch in that the high amylose corn starches contain more amylose, much more intermediate fraction and much less amylopectin than regular corn starch. For example, a high amylose type VII starch having 70% amylose by iodine titration contained 63% recrystallized butanol complex (standard amylose), 31% intermediate fraction, and only 5% amylopectin. About half of the intermediate fraction is probably low molecular weight amylose (19,000) daltons). This low molecular weight amylose fraction may be responsible for some of the rapid bond formation noted in corrugating adhesives using high amylose starches. The standard amylose determination (70%) is not fully indicative of possible performance. A better analysis would be: normal amylopectin 5%; low molecular weight, less branched amylopectin about 16%; high molecular weight amylose 63%. Total amylose is about 79%, and total amylopectin is about 21%.

Some of the true solution properties of starch appear to be related to the molecular weight of the amylose or amylopectin fractions. For example, the viscosity of jelly gums probably depends on the amylopectin molecular weight. The literature has tended to show increased molecular weights as the methods of isolating the fractions of starch and the methods of determining molecular weight have improved. Most literature indicates that amylose has a molecular weight of about 1,000,000 to 2,000,000 depending on its source, and amylopectin has a molecular weight of about 400,000,000. These are weight average molecular weights determined by light scattering experiments of the fractions with the starch dissolved in dimethylsulfoxide. Commercially used starches probably have much lower molecular weights. For example, the amylopectin of one cultivar of potato starch was consistently determined to be 65,000,000, while the amylopectin from a second cultivar was just as consistently determined to be 440,000,000. A sample of waxy maize starch had a molecular weight of 400,000,000 but after shearing a 15% solution in boiling water the amylopectin molecular weight was determined to be 10,000,000. At high molecular weights the error of measurement becomes quite large. The error at 100,000,000 is $\hat{A}\pm 10\%$, and at 500,000,000 the error is $\hat{A}\pm 20\%$. The amylopectin from pea starch appears to be the highest recorded, being 1,500,000,000 in one case.

The terms linear and branched were used in the preceding discussion in describing amylose and amylopectin. Fig. 1 illustrates the way in which glucose units are linked together to form starch. Most of the glucose units in starch are linked in a (1-4) α -D-linkage. All of the linkages are of this type in amylose (theoretically at least). This is called the linear polymer. Amylopectin contains, in addition to the above linkage, a (1-6)- α -D-linkage once in about 25 glucose units.⁴ The 1-6 linkage is called a branch point and the linear extension of this branch point is called a branch. Amylopectin is therefore called a branched polymer. Many amylases are able to hydrolyze at both the 1-4 and the 1-6 linkages, but beta amylase

hydrolyzes only at the 1-4 linkage. Further, beta amylase is able to attack only at the noreducing end of the starch molecule, and hydrolyzes off one two-glucose unit at a time only if that units is linked 1-4 (maltose). Hence, the low digestion by beta amylase (65% in some commercial amylose) suggests an occasional short branch in all except the most highly purified samples.

When starch is suspended in water it tends to increase in volume and to absorb about its weight of water. If the temperature is slowly increased there is a point at which water absorption increase dramatically. The starch granule expands 10-100 times in volume depending on the type of starch. If the amount of starch present is greater than required to absorb all of the water available, then the viscosity of the starch dispersion becomes very heavy. The swollen granules can be partially destroyed by mechanical agitation, and the viscosity will decrease, depending on the type of starch. The temperature at which the starch suddenly swells when heated in water is called the gelatinization temperature.

The unswollen starch granule is in a crystalline stated and as such is anisotropic. Most of the starch granules are birefringent under the polarizing microscope, showing a "polatrization cross." These disappear at or near the gelatinization temperature when heated in water, indicating a loss of crystallinity. With further heating the granules tend to swell a little further, collapse to a degree, and fragment if sheared to any extent. Although some starches such as waxy starch, potato starch, tapioca starch, and sago starch cook to an almost clear suspension, the starch is not in solution. This can be seen by examining the starch cook with a phase contrast or interference contrast microscope, where the swollen granules or granule fragments are evident. Most starches start to swell at about 140-1700F (60-700C), and appear to be reasonably dispersed at 2030F (950C). In order to completely solubilize the starch a much higher temperature of 300-3200F (150-1600C) is required. Some granules in the high amylose starches tend to retain their polarization crosses above 2120F (1000C), but at the 300-3200F range these granules are also completely dissolved.

The cook texture of many starches can be explained by their composition. Waxy corn starch, which is all amylopectin, when heated at 8% starch solids in water becomes very heavy and cohesive as the granules swell. With continued heating, the viscosity decreases as the granules are broken up. On cooling, the viscosity increases. The texture remains cohesive, and the solution retains its clarity. This behavior is typical when the amylopectin fraction dominates the cook characteristics. Regular corn starch, at the same solids, is much thinner, behaves like a short paste, and is quite cloudy when hot. On cooling, the cook becomes on opaque, rigid gel. In this case the amylose has modified the hot cook characteristics of the cooled cooked suspension. If the amylose has too high a molecular weight, then the gelling on cooling may not take place. Tapioca, potato, and sago starches behave more like waxy corn than corn, even though these are amylose containing starches. These amylose containing starches will set to firm gels if the starch is degraded with acid (see fluidity starches). Many of the characteristics of common starches after cooking are given in Table 2.

Table 2. Cook Characteristics of Native Starches (cooked 1 part in 15 parts water at neutral pH).

Starch	Hot Cook Body	Hot Cook Viscosity	Viscosity on Prolonged Cooking	Gel Formation on Cooling	Clarity (cold)
Corn	Short	Medium	Stable	Very high	Opaque
Wheat	Short	Relatively low	Stable	Very high	Opaque
Amioca	Stringy-cohesive	Moderately high	Thinning	Note	Fairly clear

Tapioca	Stringy-cohesive	High	Thinning	Very low	Quite clear
Sago	Stringy-cohesive	Moderately high	Thinning	Moderate	Fairly clear
Potato	Gummy, very cohesive	Very high	Thinning	Very low	Very clear

Likewise, many of the adhesive properties can be explained by reference to the amylose and amylopectin properties of the starch used. Jelly gums are usually made from waxy starches (100% amylopectin) and are stable at room temperature for many months, as would be expected from the slow retrogradation rate of this starch. A corrugating formulation must set to form a bond and become water resistant in a short period of time. The low molecular weight portion of the amylose would be expected to come out of solution fairly rapidly to make a temporary bond, and the high molecular weight portion would more slowly tend to create water resistance.

MODIFICATION OF STARCHES

The previous discussion was concerned almost exclusively with the use of native starches dispersed in water. The properties of the dispersions can be changed considerably by additives, or by modifying the starch. The principal modification in the adhesive industry is to decrease the molecular weight of the starch components so as to permit a higher solids content in the formulation. The starch is usually treated in granular form.

Fluidity Starches

Fluidity starches are made by hydrolyzing the starch in dilute acid below the gelatinization temperature of the starch. The range of fluidities is 20-90, with a 90 fluidity starch being very much thinner than a 20 fluidity. Fig. 2 gives an indication of the concentrations required to produce a given hot viscosity.

Oxidized Starches

A second modification to reduce viscosity is to treat the starch with chlorine under alkaline conditions. Lightly chlorinated starches use the water fluidity method of viscosity designation, but more heavily chlorinated products use a borax fluidity procedure. Chlorinated starches are generally called oxidized starches. They are anionic, in that they contain negative charges. This can be verified by staining with cationic dyes. Methylene Blue is a typical cationic dye.

Dextrinization

A third method of reducing viscosity is by the use of dry heat, usually in the presence of acids. This process produces dextrans. Their viscosity is also measured by using the BF or borax fluidity method. Dextrin viscosities are illustrated in Figs. 2, 3, 4. Other dextrin properties are given in Table 3.

Hydroxyethylation

Starch is treated with ethylene oxide under alkaline conditions. The major purpose is to slow down retrogradation, or precipitation of amylose from solutions of starch. This is effective because the amylose tends to be uniformly substituted, whereas amylopectin is substituted mainly near branch points.

Cationic Starches

Starch is reacted with tertiary or quaternary amine halides or epoxides under alkaline conditions. The cationic starches improve sheet strength, possibly through ionic bonding with slightly anionic paper. The cationic starches are also used in the bottle-labeling adhesives and in paperboard.

Table 3. Comparison of Dextrins.

Dry-roasting Process			
Dextrin Properties		Degree of Polymerization	
Dextrin	Acidity	Moisture	Temperature
(DPn)	Color	Swelling	Stability
White	High	High	Low
20	White	Partial	Limited
Yellow or canary	Low	Low	Moderately high
20-50	Light	High	Good
British gums	Very low-none	Very low	Moderately high to high
Very wide range	dark brown	Yellow to complete	Partial to Good

Dextrin
(DPn)
White
20
Yellow or canary
20-50
British gums
Very wide range

Acidity
Color
High
White
Low
Light
Very low-none
dark brown

Moisture
Swelling
High
Partial
Low
High
Very low
Yellow to complete

Temperature
Stability
Low
Limited
Moderately high
Good
Moderately high to high
Partial to Good

Amphoteric Starches

Cationic starches are phosphated by heating with orthophosphate to produce a starch with amphoteric (both cationic and anionic) properties. These starches offer improved pigment retention and dry strength over a wide pH range.

Miscellaneous Derivatives

These include cross linked or inhibited starches, hydroxypropylated starches, phosphorylated starches, starch succinates, grafted starches, and carboxymethyl starch. Most of the formulations using starch adhesives appear to be obtained by trial and error. The preceding listing of modifications and references is intended to enlarge the scope of possibilities for adhesive development. The discussion of the effects of amylose and amylopectin on starch dispersions serves a similar purpose

EFFECT OF ADDITIVES

Sodium Hydroxide

Sodium hydroxide (also called caustic soda) will increase tack, tend to solubilize the starch to a greater degree, increase viscosity, increase cohesiveness, and also increase color. It is usually added after cooking in water, usually to the extent of about 0.5% based on total solids. The increase in pH tends to place negative charges on the starch, which tends to explain dispersion and higher viscosity.

Borax

Borax (sodium tetraborate decahydrate) and sodium metaborate (essentially a mixture of borax and sodium hydroxide) change the properties of cooked starch dramatically. There is a large viscosity increase with increasing borax addition to about 15% (based on starch present); tack and cohesiveness are also increased greatly. Borax is used up to 10% based on starch and is usually added before cooking the starch. It acts by complexing with the starch to produce negative charges, and in addition tends to crosslink the starch. It is the crosslinking that causes the large increases in solution viscosity.

Urea

Urea is a plasticizer that acts by forming solid solutions with starch and dextrins. The urea tends to prevent crystallization of the starch or dextrin when a film is dried. It is used at 1-10% based on starch present.

Other chemicals with similar effects are sodium nitrate, dicyanamide, salicylic acid, thiocyanates, iodides,

gunaidinium salts, and formaldehyde. (Formaldehyde can also crosslink under acid conditions or be used as a preservative).

Glycerol

Glycerol acts as a plasticizer by slowing drying time, preventing excessive drying of a film. In this capacity it is a humectant.. Other humectants are ethylene glycol, invert sugars, d-glucose, and sorbitol.

Soluble Soaps

These are used as lubricants to impart flexibility regardless of atmospheric conditions. Too much lubricant will weaken adhesive bonds. Other lubricants are sulfonated castor oil and sulfated alcohols.

Urea-Formaldehyde Resin

This resin is added to give water resistance. Resorcinol-formaldehyde, poly (vinyl acetate), acrylics, and poly (vinyl alcohol) are also used to increase water resistance.

Miscellaneous Additives

Clays and bentonites are used as fillers in adhesives. Sodium bisulfite, hydrogen peroxide, sodium perborate serve as bleaches. Solvents are added to help wet water-repellent surfaces. Preservatives prevent foam during cooking. Colloid stabilizers such as soaps and sodium chloride are often added.

STARCH ADHESIVES

To be useful as an adhesive, starch must be dispersed in water, usually hot water, and a number of chemicals are added to modify the properties of the starch dispersion. The dispersion and formulation can be done by the user from the different types of starches or modifications of starches previously described. Or the user may purchase formulated adhesives from adhesives manufacturers.

Jelly Gums

These are used for bottle labeling. They are prepared by treating waxy starch or waxy fluidity starch with caustic under high shear. The starch swells in the caustic, and most of the caustic is neutralized with nitric acid. One formulation yields a final composition of 39% starch (40 fluidity waxy), 3% urea (an additive), 3% sodium nitrate (from the sodium hydroxide and nitric acid used), and 56% water (Ref. 10, p. 605). The viscosity as used is about 100,000 cP. The labels are cold water resistant.

Other Liquid Formulations

Alkaline starch formulations (prepared by adding enough caustic to gelatinize the starch) are used as carriers in corrugating, in foil-to-paper adhesives, and in carton or case sealing adhesives. If the caustic used to gelatinize the starch is neutralized, the adhesives at 18-25% starch solids are usable for wall paper printing or foil-to-paper lamination. Combinations of starches and water with added salts are the simplest adhesives, and can be used in bill posting, bag making, and tobacco seam gluing.

Pastes

Short, soft, nonstringy adhesives formulated from heavy bodied starches or dextrans are called pastes. A typical formulation for library paste is to cook a mixture of 45% low-soluble white dextrin, 5% corn starch, 5% glycerol, and 45% water.

Borated Dextrans

Dextrans are frequently formulated with borax, sodium metaborate, boric acid, or caustic in different proportions to give good tack and higher stable viscosities at moderate concentrations. The pH of the formulations is about 9.0 in most cases. Borated dextrans find use in case sealing, carton sealing, tube winding, and laminating.

White Dextrans

White dextrans are prepared by heating dry starch containing relatively large amounts of acid at low temperatures (2580F, 1200C) for rather short times (3-7 hours). Their color is white to cream, and their solubility and viscosity varies from low to high. They are used in bag-seam, tube winding, case and carton

sealing, laminating, gummed sheets, label, and envelope back seam adhesives.

Canary Dextrins

Canary dextrins are prepared by heating dry starch containing a moderate amount of acid at moderate temperatures (3000F, 1490C) for a moderate time (11 hours). They are very light to dark tan in color, have a high cold water solubility, a low stable viscosity, and excellent remoistening ability. They are used in gummed tape, envelope front seals, stamps, case and carton sealing, laminating and tube winding.

British Gums

British gums are prepared by heating dry starch with a low amount of acid at high temperature (3300F, 1660C) for a long time (17 hours). They have low to high solubilities and low to high stable viscosities, dark color, and fast tack. They are used in solid fiber laminating, bag-seam pastes, and winding adhesives.

Waxy Starch Dextrins

Waxy starch dextrins are similar to dextrins made from non-waxy starch, but have a greater viscosity stability than the corresponding regular dextrin. Envelope front seals, stamps, and gummed sheets are some uses.

Dextrin/Silicate Blends

Fast tack, rigid films, low viscosity, and very good adhesion are obtained from this combination. High-speed case sealing and fiber foil cans are some uses.

Pregelatinized Starches

Pregelatinized starches are prepared by heat swelling starch-water slurry, drying, and grinding the dry powder. The starch at 40% solids is fed to a steam-heated drum where it is gelatinized and dried, and finally scraped off the drum with a blade. These starches are used in some one-tank corrugating applications, multiwall paper bags, wallpaper, and billposting.

APPLICATION AREAS

Papermaking

Cationic, anionic, and amphoteric starch derivatives are cooked and added at the wet end of the papermaking machine at 5-20 pounds per ton of paper. The starch flocculates pigments and fine pulp particles, improves retention of fines, helps drainage, and increases internal (Scott bond) and burst (Mullen) strengths. The starches are retained by the pulp because the pulp has a slight negative charge, which holds the positively charged starch. The positive charge is present directly in the cationic and amphoteric starches, and is formed by complexing with alum in the case of anionic starches.

Unmodified starch is also used at 20-40 pounds per ton of pulp. It is retained mainly by entrapment.

Although starch is largely added at the wet end of the paper machine, it can be placed directly on the formed sheet. The starch can be sprayed onto the fibers, applied as foam directly on the wet web. These alternative methods have certain advantages, but they lose the flocculating ability of cationic or amphoteric starches.

Paper Coating

In addition to adding starch at the wet end to hold the fibers together, starch can be added at the size press, at the size press with a pigment, at the calender stack, or as a pigmented coating as a separate operation. In all of the above applications, starch acts to bind the paper fibers together or to bind the pigment particles together or to bind the pigment to the fiber. The paper has been partially dried at the size press. The viscosity of the starch solution must be relatively low (50 cP) at the size press. The 2-12% solids used in coatings would have too high a viscosity. Therefore a viscosity reduction is usually necessary. This can be accomplished by enzymes or heat treatment in the case of regular starches, or oxidized, hydroxyethylated, acid fluidity, or acetylated acid fluidity starches can be used.

Oxidized starches are anionic, and if recycled may cause pigment and fines retention problems in the wet

end. Cationic starch which has been preconverted to a low viscosity appears to have advantages in physical properties of the paper and in retention of the surface sized broke.

Starch is added at the calender stack for curl control, surface strength, laying of surface fuzz, clay coating holdout, printing characteristics, and grease or oil resistance. Solids vary from 2 to 24%, depending on the starch type used and on the paper requirements. Low viscosity starches, hydroxyethylated converted starches, and oxidized starches are used in this application.

Similar starches to those used on the size press can be used in pigment binding applications, but the viscosity of the starch should be lower. A possible formulation for a 59% solids coating is 0.2% sodium hexametaphosphate, 10.1% calcium carbonate, 40.5% clay, 8.1% low viscosity starch, 0.05% pine oil, and 0.21% soap. Thin boiling cationic starch has also been recommended for this application.¹² The same starches as are used above can be used when starch is added with pigment at the size press. Solids are generally 30-40%, and the starch to pigment ratio can be as high as 1 to 1. The viscosity is usually below 300 cP.

Corrugating

Most of the corrugated board is made using the Stein Hall system, which consists of a mixture of gelatinized starch (called the carrier starch) and ungelatinized starch. Flat corrugating medium is fluted, starch is applied to the fluted tips, and a heated liner is brought in contact with the fluted tips under heat and pressure to produce a single facer. Adhesive is applied to the fluted tips on the other side of the corrugating medium and a flat liner called a double backer liner is applied. When the facers are brought in contact with the flutes containing the adhesive, the ungelatinized starch gelatinizes. This creates an extremely high viscosity at the flute-liner interface. The applied heat also evaporates water and further increases the flute-liner bond, so that the corrugated board can be cut without delaminating. The formulation includes sodium hydroxide and borax to decrease the gelatinization temperature of the raw starch and to increase viscosity at the flute after gelatinization.

A typical old formulation is: put 13 parts water, 3.2 parts starch, 0.54 parts caustic dissolved in 0.8 parts water into a tank (called tank 1, or the upper tank), heat with steam to 1600F, agitate 15 minutes, and then add 16 parts cold water. This gelatinizes the starch present and makes the carrier starch portion of the adhesive. In another tank (called tank 2, or the lower tank) 49 parts cold water is mixed with 0.54 parts borax and 18 parts starch is added. The contents of tank 1 are slowly added to tank 2 with efficient mixing. It is possible to carry the entire operation out in tank 2, but control is more difficult.

So called no-carrier or single-component systems depend on careful control to gelatinize the correct proportion of the granules present. One formulation, which increases water resistance by adding urea-formaldehyde, is: mix 20 parts starch, 77 parts water, 0.8 parts 50% caustic, and stir at 1010F until the viscosity reaches 25 Stein Hall seconds. Then add 0.04 parts alum, 0.4 parts boric acid, and 3 parts 60% urea-formaldehyde resin. Other ways to make "no-carrier" systems are to add exactly the correct amount of caustic and stop the swelling by carefully injecting steam into the mixture; or partially swell the granules by milling a 10-40% moisture starch to damage granules before suspending in water. A chemical-mechanical method of producing the carrier starch is to introduce a mixture of 12% starch and 30% caustic into a centrifugal pump. Final alkalinity was 14.4% and the viscosity was 4200 cP. at 760F. The use of starches having different gelatinization temperatures is given in Ref. 14. Here 350 parts tapioca starch and 3000 parts corn starch are suspended in 9000 parts water and treated with 500 parts of borax are added. The Stein Hall viscosity is 52 seconds, and the gel point is 630C (1450F). The tapioca starch swells before the corn starch, and essentially produces the two-component system.

High amylose starches improve water resistance and increase speed of corrugation. A formula for the carrier starch is: 1192 parts water, 424 parts high amylose starch, 6 parts borax: bring to 1300F (540C) and add with stirring a mixture of 36.6 parts caustic in 47.5 parts water. The raw starch portion is made by

mixing 3480 parts water, 1600 parts corn starch, 28 parts borax and 91.2 parts thermosetting resin. Mix the carrier into the raw starch.

It appears, at least in the case of high amylose carrier starch, that the carrier starch is the principal adhesive. The raw starch, when gelatinized, absorbs water to concentrate the dispersed high amylose starch on the flutes. Older ideas of the mechanism were that the carrier starch was simply a suspending agent for the raw starch, which was the principal adhesive. One suggestion was that amylose migrating out of the granules caused at least the initial tack.

There are many publications on the use of high amylose starches. High amylose starch is esterified with acetic anhydride or succinic anhydride to improve stability. Another patent concerns the addition of dihydroxyethyleneurea together with acetone-formaldehyde to improve water resistance.

The use of high amylose starches in the production of cold corrugating adhesives (not requiring extensive heating) has been published. A mixture of oxidized and hydrolyzed 70% amylose containing starches is dispersed as a 35% starch suspension, gelatinized at 140°C, and used in corrugating without steam. The high amylose starch was treated with 4% sodium hypochlorite at an initial pH 11 to make part A of the degraded mixture. Part B was made by hydrolyzing high amylose starch for 12 hours at 50°C (122°F) with 6% of 35% hydrochloric acid. Both A and B were neutralized to pH 5, washed, filtered, and dried. Then 70% A and 30% B were mixed to a 35% suspension and cooked at 140°C. The use of the B portion is claimed to improve speed from 90 m/min to 230 m/min.

Mixtures of waxy, regular, and high amylose starches are degraded with a mixture of sodium persulfate, sodium sulfate, boric acid, and caustic, and cooked at 90°C (194°F) and about 33% solids to form a starch adhesive that hardens on cooling. The cold corrugating process is described in other references.

Other claims for improving corrugating formulations include increasing the carrier starch solids, addition of urea, using crosslinked starch in the carrier, and using cationic starch as the carrier starch.

Bag Adhesives

Three adhesives are used in paper bag manufacture: side seam adhesives, bottom paste adhesives, and cross pastes. The side seam adhesive is used to form a cylinder from a flat sheet of paper. This adhesive must develop a strong bond quickly, so the tube can be cut for further operation. The viscosity should be about 3,000 cP, and solids about 25%. One formulation is: water 68%; heat to 160°F (71°C), add 3% preservative.⁴ A water-resistant formulation is: water 1700 pounds, white dextrin 700, soap 2, urea-formaldehyde 70, heat at 200°F (93°C), dilute with cold water to 260 gal volume, and add 14 pounds ammonium chloride. The formulation should be used immediately. Its pH is about 6.

Bottom paste adhesives are applied to one end of the tube formed above to close that end to form a bag bottom. These pastes are usually made from unconverted starches. Soap and/or salt may be added to produce a thixotropic paste (flows under shear but sets up if left undisturbed). A water-resistant formulation is corn starch 13%, poly (vinylalcohol) 4.5%, poly (vinylacetate) 1%, soap 0.1%, water 81%, heat to 90°C (194°F), cool to room temperature.¹⁰

Cross paste is used for multiwall bags to glue the plies together before forming a tube. This is similar to seam paste, but should not penetrate the ply. Clay or poly (vinylacetate) is added to prevent penetration.^{10,11} The seam adhesive for multiwall bags can be somewhat heavier than given above, and higher molecular weight white dextrans can be employed. The bottom adhesive is usually a fluidity starch. A mechanical way of converting potato starch for use in paper bags is also described.²⁹

Laminating Adhesives

The requirements of the particular equipment must be met for bonding paper to paper or to paperboard, for making poster displays, bonding paperboard to paperboard, or rotary lamination. Lay-flat, or noncurling, is an important attribute of these adhesives. High tack and low penetration are also required. One formulation is water 43%, high soluble white dextrin 21%, corn starch 4%, sodium nitrate 32%; heat to 200°F (93°C),

hold 20 minutes, add preservative. Another formulation is high soluble white dextrin 20%, clay 13.5%, urea 6.7%, borax 5%, water 55%.¹⁰

Foil laminations usually call for resins, but even here a small amount of starch is often added for its smoothing properties. One example is: poly (vinylalcohol) 3, starch 3, water 49, potassium persulfate 0.1; add dropwise a mixture of 5 dibutyl phthalate and 39 vinyl acetate at 700C (1580F), dilute to 25% solids. Starch at 3-15% is claimed to prevent coarse particle formation in the following formulation: 45% aqueous emulsion containing 1:99 acrylic acid: vinyl acetate copolymer 100, corn starch 4, poly (vinylalcohol) 5, dibutyl phthalate 1.5 Aluminum foil was coated on paper at 300 m/min (meters per minute) without coarse particle formation, while a similar formulation omitting the starch formed coarse particles at 90 m/min.

Tube Winding

Tube winding is either spiral (a continuous winding where adhesive is applied to outer plies as they are wrapped on a cylindrical mandrel), or convolute (where the sheet is as wide as the mandrel is long, and the mandrel wraps the sheet over itself). The tube is removed from the mandrel by a pusher arm. Convolute adhesives are usually used cold, while spiral adhesives may be used at 1310F (550C). Many starch products may be used, but 50% solids borated dextrans are common.

Corrugated Boxes

The tops and bottoms of corrugated boxes are closed with case sealing adhesive. Although liquid glues and hot melts are preferred, carton adhesives are used, sometimes with added caustic. Carton sealing involves bonding the bottom and top flaps of folded paper boxes. A top bottom carton sealing adhesive is water 51%, white dextrin 37%, preservative 1%, borax 6%, antifoam 0.06%; cook to 1850F (850C) for 20 minutes, cool to 1200F (490C), add 5 water and 0.6 of 50% caustic.

Gummed Tapes

The types of remoistenable tapes are regular sealing tapes, reinforced sealing tapes, and box tapes. A regular sealing tape formulation is: thin boiling waxy sealing tape formulation is: thin boiling waxy starch 39.5, canary dextrin 17, polyacrylamide 2, dispersing agent 0.4, water 41.1 is suggested.¹⁰ Box tape is usually made from animal glue, but one patent claims performance superior to animal glue from a starch acrylamide graft copolymer prepared as follows: water 51%, sodium nitrate 7%, waxy corn starch 33%, copper sulfate 0.03%, acrylamide 10%; mix, then add 0.05% ammonium persulfate and 0.03% sodium metabisulfate; heat rapidly to 2000F (930C) and maintain for 15 minutes. Add sodium tartrate to adjust pH to 5.5.

The use of an oxidized starch acetate 180, urea 20, and water 200 as a gummed tape adhesive has been patented. The formulation of an acrylamide starch graft polymer is also claimed: hydrolyzed low viscosity acrylamide-starch graft copolymer 67, canary dextrin 20, animal glue 10, urea 10, petrolatum 0.25, and sodium hexametaphosphate 0.1, are heated for 3 minutes at 185-2050F (85-960C).

Label and Envelope Adhesives

The general adhesives for these uses can be found in Table 4. Dextrans, enzyme-converted starches, or mechanically degraded starches are suitable, with white or canary dextrans made from tapioca, waxy corn, and regular corn starches the most used. The envelope front seam is remoistenable and can have 55-65% solids with a viscosity of 2,000-10,000 cP. After drying the adhesive should be non-blocking at high humidity. This requires a nonhygroscopic plasticizer. One formulation containing poly (ethylene glycol) is high soluble waxy corn dextrin 63, sodium bisulfite 1, Carbowax 4,000 (Union Carbide) 0.5, water 35.5. Backseam adhesives can be a little thinner with viscosities close to 1000 cP, and 40-50% solids.

Discrete particle, flat gumming, solvent-based adhesives are another method of making remoistenable adhesives. The cold-water-soluble material (usually a dextrin) is suspended in a solvent and applied to the paper and dried. Since the dextrin is not soluble in the solvent, it is deposited in discrete particles. On contact with water, the dextrin swells and will glue two pieces of paper together. The dextrin is usually used

at 40% solids and a viscosity of 1000 cP.

Paper Box

Paper box manufacture involves the operations of ending (box body formation), stripping (coating the box walls with precut wrap and smoothing), tight wrap (machine wrapping a box with glue on most of the wrap), and loose wrapping (adhesive only on border of the wrap). Borated dextrans containing 10-15% borax and cooked at 30-50% solids are usually used. See Table 4 for more information.

Textiles

Starch is used predominantly in warp sizing in the textile field. The purpose is to strengthen the yarns for weaving operations. A typical formulation is corn starch 8.5%, softener (tallow or sulfonated oil) 1.5%, kerosene 0.2%, cook to 1900F (880C), and homogenize at 2000 psi. Oxidized starches are used in finishing and printing, and low treatment starch acetates, starch monophosphate blended with poly (vinyl alcohol), cationic and amphoteric starches, as well as fluidity starches are used in finishing and warp sizing.

Wall Covering Adhesives

These require good wet tack and good slip. Plasticizers give slip (the ability to move the paper around to obtain a good fit), borax gives tack, and clay allows easier removal of the paper later. A formulation consists of acid modified pregelled starch 25, clay 20, urea 3.75, sodium metaborate 1.25, and water 50. A repolymer is: dry blend carboxymethyl cellulose 25, hydroxyethyl cellulose 8.5, sodium alginate 0.5, anionic polyacrylamide 0.3, urea 30, potato starch 27, colloidal silica 1, sodium stearate 2.5, sodium soap 3.5, sodium fluoride 0.5, and 4-chloro-meta-cresol 1. The mixture is suspended in 3 parts water, coated on paper and dried. A second formulation is: starch 375, carboxymethyl cellulose 400, urea 200, polyacrylamide 5, colloidal silica 5, parachloro-meta-cresol 10, sodium fluoride 5. An inverted emulsion formulation containing a starch graft copolymer has also been suggested for wallpaper adhesives.

Miscellaneous Uses

Gelatinized starch 100, dextrin 100 is used to make fish good stable in water. A 1:1 mixture of starch and polyacrylamide was used to make an adhesive for high voltage transformers. A mixture of poly (vinyl acetate) emulsion 100, plasticizer 20, and starch 20 has been used in book binding.

GOVERNMENTAL REGULATIONS: ADDITIVES

The Food and Drug Administration (FDA) of the U.S. Department of Health, Education and Welfare (HEW) has issued a series of regulations concerning the use of potentially toxic substances in packaging materials contacting food. Most of the regulations may be found in the Code of Federal Regulations, Title 21, revised annually (21 CFR 121.2520, "Adhesive").

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