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ECONOMICS OF PRODUCTION OF SYNTHETIC RESIN ADHESIVES ^{1/}

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ECONOMICS OF PRODUCTION OF SYNTHETIC RESIN ADHESIVES

1 Introduction

Synthetic resins were introduced as adhesives in the wood processing industry in the early 1930-s. The War years saw great progress in their development and use. Their consumption is still increasing.

Synthetic resin adhesives for wood may be thermosetting or thermoplastic. A thermosetting resin is one that undergoes crosslinking upon hardening and becomes substantially infusible and insoluble. The crosslinking or hardening reaction is catalysed by heat or chemicals and is irreversible. The most important thermosetting resins for adhesives are urea-, melamine-, phenol-, and resorcinol-formaldehyde. About 1.7 million tonnes of urea formaldehyde and phenol formaldehyde resins were used in the plywood and particle board industries in 1971 and this figure is expected to double by 1980. The resin adhesive accounts for 5-50 per cent of the cost of the finished panel depending on the grade and proportion of adhesive used.

Thermoplastic resins do not normally undergo any crosslinking during curing or hardening but remain in a reversible state and can soften on subsequent heating. The most important thermoplastic resin for wood adhesive is polyvinyl acetate resin.

The raw materials from which these resins are made are based almost entirely on crude oil and natural gas. Synthesis of the

raw materials from coal and air is possible. Small quantities of phenol and cresols are obtained from coal at present by distillation.

2 Production of Synthetic Resins for Wood Adhesives

2.1 Urea formaldehyde (UF) resin

Urea formaldehyde resins commonly used as wood adhesives are products of chemical condensation of urea and formaldehyde. Urea is a white crystalline powder melting at 132.6°C. The technical grade containing about 46% nitrogen is normally used for resin manufacture. Formaldehyde is a gas usually available as a solution of 30 to 37 per cent concentration (by weight) in water with methyl alcohol as stabilizer. The solution is known as formalin. The reaction of urea and formaldehyde is complicated and a series of reaction products of different chemical composition are formed. The first step in the reaction between urea and formaldehyde is the formation of methylolureas. Up to 4 formaldehyde molecules can react with one molecule of urea but for wood adhesives reaction conditions which lead mostly to the formation of dimethylolurea are employed.

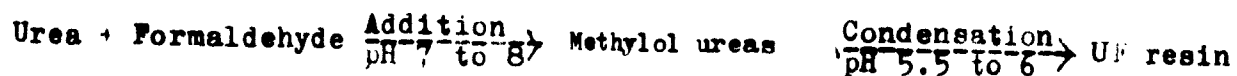
The rate at which methylolurea formation takes place depends on temperature, pH and ratio of reactants. To control the reaction and to favour the formation of dimethylolurea, the conditions must be slightly alkaline and a pH of 7 - 8 is employed.

The next step is the condensation of the monomeric methylolurea to form polymer molecules. The reaction is carried out at 95-100°C and the reaction speed is controlled by the acidity of

the medium. Best results are obtained at a pH of 5.6 - 6. As the condensation proceeds larger molecules with crosslinked structure are formed. If the reaction is allowed to continue the crosslinking will lead to gelatinization of the resin in the reaction kettle. The condensation must therefore be closely watched and controlled at all stages of production. The different steps in the formation of the resin are shown in Table 1.

Table 1
Steps in the formation of urea formaldehyde resin

Resin	Raw material	State	Mole ratio U/F	Reaction	Semi-product	Reaction	Product	Final pH
UF	Urea	White crystalline powder		Addition	Monomethylol urea and dimethylol urea	Condensation	UF resin	7.5-8.0
	Formaldehyde	Gas dissolved in water	1 : 1.5-2.0					



A large number of UF resins can be made and are in fact being manufactured for the wood industry. Resins are made as aqueous dispersions of different solid contents and also as spray dried powders. Molar ratios of formaldehyde to urea in the range of 1.5 to 2 are employed.

2.1.1 Manufacture of urea formaldehyde resin for plywood

A schematic diagram of the equipment used for resin manufacture is shown in Figure 1. It consists of a reactor or kettle(1),

constructed of stainless steel, which is provided with a stirrer(2), jacket(3) for steam heating and water cooling, an inlet(4) for the reactants, a pressure relieving valve(5) and a sight glass(6) for visual observation. A condenser(7) which may be operated either under vacuum or at atmospheric pressure is provided for refluxing the volatile constituents. The stirrer is coupled to a constant speed motor(8) through a reduction gear. The jacket is provided with an inlet(9) and an outlet(10) together with a drainage vent(12). The temperature of the reaction mixture is measured by probe(11) and is connected to an electronic relay for semiautomatic control. While the emptying of the kettle is done through the main outlet(13), sampling of the contents of the kettle during reaction is carried out with the aid of the sampling chamber(14). After the completion of the reaction, the liquid resin is pumped to the storage vessel(15) by means of a gear pump(16).

1500 kg formalin (about 37%) is charged into the resin kettle. pH of the formalin is raised to 7.2 to 7.5 by the addition of caustic soda solution (30 to 35% concentration). 600 kg urea is added and mixing is carried out to get a clear solution. The solution is heated to boiling and kept boiling for 20 - 30 minutes to complete the formation of dimethylolurea, care being taken to maintain the pH at 7.2 to 7.5.

In the next stage, the solution is made acidic (pH 5.6-6.0) by the addition of an acid such as formic acid, acetic acid,

citric acid, lactic acid, and phosphoric acid of 10-15% concentration. Water is removed by distillation under atmospheric pressure when the condensation reaction also proceeds. After removal of the calculated quantity of water (250 kg) the kettle contents are refluxed. Condensation is stopped when the viscosity of the resin in the kettle reaches about 500 cp or the water tolerance is about 4 to 6 times. Viscosity may be determined by a bubble viscometer. Water tolerance is the number of times the resin solution can be diluted before incipient precipitation is observed. The progress of the reaction is continuously checked by these methods to know when the desired degree of condensation has taken place. In the absence of bubble viscometer a flow cup may also be used.

The resin syrup in the kettle is again made alkaline by the addition of caustic soda solution (30-35% concentration) to pH 8 so that further condensation is stopped. The resulting resin syrup has a concentration of about 55 per cent and may be cooled to room temperature by cold water circulation and discharged for formulation of adhesives. The yield is about 1800 kg.

To obtain resin syrup of higher concentration the contents of the kettle, on completion of the condensation reaction to the desired stage, are cooled to about 60 - 65°C by cold water circulation and distilled under reduced pressure to remove 150 kg water. A liquid adhesive of about 66 per cent solid content is obtained. It may be cooled to room temperature and discharged. Yield is about 1600 kg. The liquid UF resin has a storage life

of about 1 - 6 months depending on temperature of storage. The manufacture of the resin syrup takes 4 - 8 hours depending on the size of the charge and procedure adopted.

The UF resin syrup can be epray-dried to obtain powder resin which can be transported easily and has a longer storage life (shelf life) of 6 to 18 months. The resin, whether syrup or powder, should be stored in a cool dry place.

2.1.2 Manufacture of urea formaldehyde resin for particle board

Urea formaldehyde resin for particle board manufacture is essentially monomeric. The low degree of polymerization is necessary for solubility of the resin for ease of spray application. UF resin for particle board is therefore characterized by a lower proportion of formaldehyde than in plywood resin. This also favours reduced release of formaldehyde from particle boards.

To manufacture UF resin for particle board, the same procedure as for the first stage in the manufacture of UF resin for plywood is followed. 200 kg urea is now added at reflux and refluxing continued for 30 minutes, the pH being maintained at 7.2 - 7.5 throughout the reaction. The resulting resin of solid content about 59 per cent may be cooled and discharged.

If a resin of higher solid content is desired, the resin is cooled to 60-65°C and 200 kg water is removed under reduced pressure to obtain a resin syrup of about 65 per cent concentration.

The viscosity of UF resin for particle board is in the range of 100 - 500 cp. UF resin for plywood (Section 2.1.1) may also be used for particle board manufacture. Five per cent urea on the dry weight of the UF resin should be added along with the hardener to the adhesive mix.

2.2 Phenol formaldehyde (PF) resin

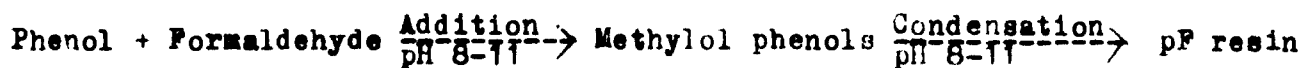
Phenol formaldehyde resin is manufactured from phenol and formaldehyde. Phenol in pure condition is a white crystalline solid melting at 43°C. The reaction of phenol and formaldehyde is rather complicated and a number of reaction products of different compositions are formed depending on the reaction conditions. The first step in the reaction is the addition of 1 to 3 molecules of formaldehyde to phenol to form methylol phenols. The average number of methylol groups per phenol molecule will depend on the mole ratio of formaldehyde to phenol. The ratio is usually 1.3 to 2.2 for resins for the wood industry.

The formation of methylol phenols and their further interaction to form crosslinked structures take place in alkaline medium (pH 8 - 11), the speed of reaction increasing with pH. The reaction is highly exothermic in nature and requires to be controlled with the utmost care to prevent gelling of the resin in the reaction kettle. The different steps in the formation of the resin are shown in Table 2.

Table 2

Steps in the formation of phenol formaldehyde resin

Resin	Raw material	State	Mole ratio P/F	Reaction	Semi-product	Reaction	Product	pH of the resin
PF	Phenol	White crystalline solid	1:1.0-3.0	Addition	Methylol phenols	Condensation	PF resin	8 - 11
	Formaldehyde	Gas dissolved in water						



2.2.1 Manufacture of phenol formaldehyde resin

The resin kettle used for manufacture of PF resin is similar to that used for UF resins (Figure 1). The kettle may however be of mild steel.

400 kg molten phenol at 60 - 65°C is charged into the kettle followed by 600 kg formaldehyde (37% concentration). Stirring is started and is continued throughout the cycle. 32 kg sodium hydroxide dissolved in 65 kg water is then added. Steam is sent through the reactor coils to raise the temperature (to about 60°C) until the exothermic reaction becomes strong enough to cause the batch to heat up without further steam. The temperature is maintained at about 60°C by circulating cold water. Control over the reaction is critical and must be carefully exercised both for uniformity of product and the avoidance of too much crosslinkage which would result in gelling of the resin in the kettle. After the vigorous exotherm, condensation reaction may be continued in the temperature range of 60 to 65°C for 4 to 5 hours or 85 - 90°C

for 60 to 90 minutes. When the viscosity of the resin syrup is about 100 cp and the water tolerance is about 5 times, the resin is cooled to room temperature by circulating cold water and discharged from the kettle. Yield of resin syrup is about 1000 kg and solid content about 50 per cent.

The storage life of the resin, which depends mainly on the storage temperature, is 1 - 3 months at 20 - 25°C.

PF resins are also produced in powder form by spray drying the syrup. The resin is also available in film form, impregnated on tissue paper for use as adhesives.

2.3 Resorcinol formaldehyde (RF) and Phenol resorcinol formaldehyde (PRF) resins

Resorcinol is a crystalline solid melting at 110.7°C. It is a very highly reactive phenol so that RF resins cure at room temperature. Because of its high reactivity RF resins are prepared with only a part of the formaldehyde required for complete cure the balance being added at the time of application of the resin adhesive. Whereas a PF resin requires very strong acid catalyst for curing at room temperature, RF and PRF resins cure at nearly neutral pH (6.5 - 7.0 for RF and 7.0 - 7.5 for PRF).

Resorcinol is a costly chemical. To reduce the cost of the resin, part of the resorcinol in RF resin can be replaced by phenol.

RF and PRF resins are mostly used as special purpose adhesives in the assembly gluing of wood and wood products. These adhesives withstand exposure to weather better than most timbers themselves. They comply with the highest requirements of all specifications and have outstanding durability under the most severe conditions.

2.3.1 Manufacture of resorcinol formaldehyde resin

10.0 kg resorcinol is dissolved in 8.0 kg water in a resin kettle. 5.0 kg formalin of 37 per cent concentration is added and the pH adjusted to 7.0 to 7.5 with 40 per cent sodium hydroxide solution. The mixture is stirred and heated to 80 - 85°C and maintained at that temperature until the desired polymerization is reached, which is determined by resin viscosity (100 - 200 cp). The time required for this cycle is about 1 hour. The resin is cooled thoroughly to room temperature and stored. Storage life of the resin is about 1 year.

2.3.2 Manufacture of phenol resorcinol formaldehyde resin

10.0 kg of molten phenol is charged into a reaction kettle followed by 18.0 kg formalin (37%). The pH is adjusted to 9.5 - 10 by adding 0.60 kg sodium hydroxide dissolved in 1.2 kg water with continuous stirring. The mixture is maintained at 70 - 75°C until the desired polymerization is reached which is determined by viscosity (50 - 100 cp) and water tolerance (about 1 : 12). The temperature is brought down to 50 - 60°C by circulating cold water. 10.0 kg resorcinol is added slowly with constant stirring and condensation is continued at 70 - 75°C to get the required degree of polymerization which is measured by viscosity (about 200 cp) and water tolerance (about 1 : 6). The resin is cooled to room temperature by circulating cold water. The resin obtained has a pH of 7.2 - 7.5 and has a storage life of about 2 - 3 months at 25°C.

2.4 Melamine formaldehyde (MF) resin

Melamine is a white crystalline powder melting at 250°C. The reaction between melamine and formaldehyde is similar to that between urea and formaldehyde. A range of MF resins are made with a molar ratio of 1:2 to 1:3.

The reaction is carried out in the alkaline medium at a pH of 8 - 10. Condensation at the lower pH gives a polymer that is more hydrophobic and less stable than the resin obtained at the higher pH.

2.4.1 Manufacture of melamine formaldehyde resin

100 kg formalin (37%) is taken in a resin kettle and is made alkaline (pH 9) with aqueous sodium carbonate. 50 kg melamine is added to the kettle and the mixture is heated to 80 - 98°C for 1 - 2 hours with continuous stirring. The reaction is stopped by circulating cold water when the water tolerance of resin is about 4 times. The cooled resin syrup is discharged from the kettle. Yield about 140 kg. Storage life about 2 - 3 weeks at 25 - 30°C.

Melamine resin prepared in the spray dried form has a storage life of about one year.

2.5 Epoxy resin

Epoxy resins are thermosetting and are so called because they contain in their structure the reactive epoxide group $\text{>C} \begin{array}{c} \diagup \text{O} \diagdown \\ \text{---} \end{array} \text{C} \text{<}$ Both basic and acidic materials react with the group and depending on their functionality give crosslinked infusible and irreversible products. Epoxy resins harden at room temperature without the

production of volatile byproducts, so that shrinkage of the resin is low and joints may be made with only the minimum pressure necessary to bring the surfaces together. Cohesive strength of the cured resin often exceeds adherend strength. Hardeners include aliphatic polyamines, polyamides and polysulphides.

Most commercial epoxy resins are prepared by the reaction of bisphenol A [2,2-bis (4'-hydroxy phenyl) propane] and epichlorohydrin (1-chloro-2,3-epoxypropane), but other types of products are becoming increasingly available.

2.5.1 Manufacture of epoxy (bisphenol A-epichlorohydrin) resin

The principal raw materials required for resin production are bisphenol A and epichlorohydrin.

In a typical process for the preparation of a liquid epoxy resin, a mixture of bisphenol A and epichlorohydrin (about 1:6 molar) is heated to about 60°C with stirring. Solid sodium hydroxide (2 moles per mole of bisphenol A) is added slowly. The reaction is exothermic and cooling is applied to keep the temperature near 60°C. Excess of epichlorohydrin is then removed by distillation under reduced pressure (50 mm of Hg). Toluene is added to the residue to precipitate the salt present which is filtered off. The toluene is removed by distillation under reduced pressure and the resin is heated up to 170°C at 25 mm of Hg pressure to remove traces of volatile matter.

2.6 Polyvinyl acetate (PVAc) resin emulsion

The principal raw material for the manufacture of PVAc is vinyl acetate monomer. In commercial practice emulsion polymerization is the predominant method, the resulting latices being used directly for adhesives.

2.6.1 Manufacture of polyvinyl acetate resin emulsion

Polymerisation of vinyl acetate is normally carried out batchwise in a stirred reactor, jacketed for heating and cooling.

A typical formulation is as follows.

Vinyl acetate (inhibitor free)	..	100 parts (by weight)
Water	..	100 parts
Protective colloid	..	2.5 parts
Surfactant	..	0.1 parts
Sodium bicarbonate (buffer)	..	0.5 parts
Potassium persulphate (initiator)	..	0.5 parts

Reaction temperature is about 75 - 80°C and reaction time is about 2 hours. The reaction is highly exothermic and in order to achieve better control and a product with smaller particle size only a portion of the monomer is initially taken in the reactor and when this has polymerized the remainder is added slowly over 2 to 4 hours. The resulting latex is used for formulating wood adhesives.

3 Features of Fillers, Extenders and other Additives

3.1 Fillers and extenders

Fillers are relatively inert substances added to an adhesive to improve its working properties, permanence, strength or other qualities. These materials when added to synthetic resin adhesives improve their spreading characteristics on conventional roller spreaders and also control excessive adhesive penetration into porous woods. Many different materials, both organic and inorganic, are used as fillers. Examples are wood flour, coconut shell flour and walnut shell flour, among organic materials and gypsum, chalk and china clay among inorganic materials.

An extender is a substance, generally having some adhesive action, added to an adhesive to reduce the amount of the primary binder required per unit area, thus reducing the cost of the joint. The three main requirements of an extender are (i) It should readily disperse in the liquid glue giving a smooth and uniform glue mix which maintains its viscosity during its application. (ii) It should improve the bonding capacity of the glue and increase the pot life. (iii) It should help to spread a specific quantity of glue solids without excessive dripping or squeeze out.

Various types of extenders are in use at present. For example (1) Starchy materials, such as tapioca starch, potato starch, maize flour, wheat flour, other cereal flours, tamarind seed powder, etc. (2) Proteinous materials such as casein, groundnut (peanut) meal, groundnut protein, maize gluten, soyabean meal, blood albumin, animal glue, etc. and (3) Other materials such as defatted sal meal, tannin extracts, lignin, etc.

Extenders may also serve the purpose of fillers. It has therefore been the practice to use the terms filler and extender rather loosely and often synonymously.

The amount of filler or extender that can be added to an adhesive varies considerably, depending on the density, particle geometry and chemical composition of the material. A small particle size (preferably -150 to -200 mesh) is necessary in order to ensure a smoothly spreading mix. Proteinous extenders which can be dispersed in alkali may be coarser (-100 mesh).

Extenders are often used in high ratios, up to 100 per cent or more, while fillers normally are limited to 10 to 25 per cent. High proportions of extenders in adhesive are possible only if the materials are very finely ground or dispersed. The choice of extender or filler depends on the type of adhesive and also the end use of the glued product envisaged. Some of the extender materials are human or animal food which is a factor to be considered in the choice of extenders.

Among the most useful extenders used with UF glues especially in plywood manufacture are starchy and proteinous materials. Extenders containing starch give excellent results when hot pressed. Water causes starch to swell. If the temperature of hot pressing is high enough the swelling continues until the granules burst and form a colloidal dispersion. This transformation known as gelatinization takes place within the normal curing temperature of a hot setting adhesive. If the curing temperature of the adhesive is below the gelatinization point starch behaves predominantly as an inert filler and contributes little towards adhesion properties. If starches are gelatinized it assists in preserving the viscosity of the adhesive as the temperature rises, and also contributes to adhesion. For room temperature curing adhesives starch which has been chemically processed or gelatinized in hot water and cooled may be employed.

Large additions of extenders make UF and PF resin adhesives sensitive to moisture and susceptible to attack by micro-organisms. About 25 per cent is considered the safe limit.

The greater the quantity of extender added, the heavier the glue spread must be to ensure best results. Similarly, in some cases pressing times are lengthened by the addition of extenders. The choice and the proportion of extender will be governed by the cost of the resin and extender as well as the effect on glue spread rate, press time, press temperature, etc.

3.2 Solvents and thinners

Solvents are needed in most adhesives to disperse the binder to a spreadable consistency. In most wood bonding adhesives the solvent is water. For reconstituting PF resin powder some alcohol is used together with water. Demineralised water is to be preferred, although any potable water may be used.

Thinners are volatile liquids added to an adhesive to modify the consistency or other properties. They are not ordinarily employed with wood bonding adhesives.

3.3 Plasticizers

A plasticizer is usually a high boiling liquid or low melting solid incorporated in an adhesive to increase its flexibility and workability. An example is the addition of dibutyl phthalate to polyvinyl acetate resin emulsion. The addition of a thermo-plastic resin such as polyvinyl butyral or an elastomer to PF resin for bonding metal to wood is an instance of plasticization using a polymeric material. Plasticization can also be imparted by copolymerization. Examples are the condensation of a mixture of phenol and cresols (ortho and para) or phenol and cashewnut shell liquid with formaldehyde in the manufacture of PF resins which are less brittle than straight PF resin.

3.4 Catalysts

Catalysts are substances which speed up the rate of curing reaction of an adhesive when added in small proportions. A good example of a catalyst is ammonium chloride commonly used to speed up the curing of UF wood adhesives. The proportion of catalyst to resin is adjusted to provide a practical working life for the adhesive mix while ensuring complete cure of the resin. The catalytic action may be further controlled by the addition of modifiers. For example buffers or retarders such as ammonia, tricalcium phosphate, zinc oxide, urea and melamine may be added to control the acidity of catalysts added to UF resin.

3.5 Hardeners

Hardeners are substances added to an adhesive to promote or control the curing reaction by taking part in it. Hardener is therefore a reactant in the curing reaction. A good example of a hardener is paraformaldehyde commonly used with RP or PRF resin adhesive.

In the wood working industry the terms catalyst and hardener are often used synonymously, e.g. ammonium chloride catalyst for UF resin is often referred to as hardener. Mixtures of catalyst, modifiers of catalyst and fortifiers may also be termed as a hardener in the industry. An example is a mixture of catalyst, modifiers and melamine fortifier used with UF resin adhesive.

3.6 Fortifiers

Fortifiers are materials which are added to an adhesive to improve the strength, moisture resistance and durability of the

resultant bond. They usually have some distinct adhesive value. An example is melamine or melamine formaldehyde resin added to UF wood glue to improve its water resistance and durability. Tannin extracts of mimosa and quebracho may be used as fortifiers to improve the water resistance of UF adhesives. PF resin is used as fortifier in mimosa tannin formaldehyde plywood adhesives to obtain high wood failure in tests of the joint. A fortifier may also sometimes function as an accelerator.

3.7 Preservatives

Preservatives are added to certain adhesives to prevent or retard decomposition by micro-organisms either while the adhesive is being stored and applied or during service of the completed bond. Preservatives which include inorganic materials such as copper salts and organic compounds such as chlorinated phenols are most important in formulations containing starch or proteins. Preservatives, usually chlorinated hydrocarbons, are added to certain wood adhesives to protect wood from insects. For plywood intended for packing foodstuff such as tea, borax and/or boric acid is used.

4 Compositions of Synthetic Resin Adhesives

4.1 Formulation of adhesives

The synthetic resins mentioned in section 2, usually require compounding with one or more additives such as fillers, extenders, catalysts, hardeners, fortifiers and preservatives to obtain adhesives for bonding wood. Resins which are in powder form have to be dispersed in water for mixing with the additives and for application.

Adhesives may be room temperature setting or hot setting. They can be compounded to satisfy specific needs with regard to quality of bond, species of wood and manufacturing conditions. It is important to weigh or measure all the ingredients of an adhesive carefully before mixing them together.

Adhesives for wood may be classified in different ways. Classification by resistance to moisture is often made use of. In Indian Standard 303-1975 Specification for general purpose plywood, four classes of adhesive bond are specified. In ascending order of durability, these are cold water resistant (CWR), warm water resistant (WWR), boiling water resistant (BWR) and boiling water proof (BWP). This classification corresponds approximately with the classification in British Standard 1455- August 1972 Specification for plywood manufactured from tropical hardwoods, where adhesive bonds are classified as interior, moisture resistant, boil resistant, and weather and boil proof. Table 3 attempts a comparison of a few national standards.

Table 3

Comparison of durability category and chemical type of adhesive in some national standards.

Country and Specification number	India	U.K.	U.S.A.	Japan	Germany
	IS:303	BS:1455	CS 35-63	Export plywood standard	DIN-68705
Most durable: Phenolic.	BWP	Type-WBP	Type-I	Type-I	Type AW-100
Durable: Phenolic extended.	BWR	-	-	-	-
UP fortified with MF.	BWR	Type-BR	-	-	Type A-100
Good water resistance: Less resistant to prolonged wetting and drying:					
UP with some filler.	WWR	Type-MR	Type II	Type II	Type IW-67
UP with extender fortified with melamine.	WWR	-	-	-	-
Resistant to cold water for short periods:					
Heavily extended UP.	CWR	Type-INT	Type III	Type III	Type IP-20

Cold water resistant (CWR) adhesives. This group of adhesives include UP resin adhesive which has been heavily extended. The resulting bond is not suitable for exposure to weather. Even in interior protected locations it has a relatively short life if the ambient temperature is high and there are sharp fluctuations in humidity. This class of adhesives should be used for plywood manufacture only when the plywood is intended for use in interior protected locations for relatively short periods.

Warm water resistant (WWR) adhesives. This class of adhesives include UF resin with a filler. Joints made with these adhesives have the ability to resist immersion in water for three hours at 70°C. Joints with WWR adhesives will give long service in interior protected locations but are not suitable for outdoor exposure or to periodic wetting and drying. It is sometimes economical to use a relatively higher proportion of extender together with a fortifier for the resin in this class of adhesives.

Boiling water resistant (BWR) adhesives. This class of adhesives include (1) UF resin fortified with resorcinol or melamine formaldehyde resin and (2) PF resin with extender. Wood joints made with these adhesives may be described as semi-exterior grade and can be exposed for short periods. In interior locations in a tropical environment the joints will give good service.

Boiling water proof (BWP) adhesives. These adhesives consist of phenolic resins including RP and PRF resins and may contain small proportions of filler but no extender. Tannin formaldehyde adhesives fortified with PF or PRF resin may also belong to this class. Wood joints made with these adhesives are durable in outdoor exposure situations.

4.2 Formaldehyde adhesives

4.2.1 Formaldehyde adhesives for plywood

4.2.1.1 Urea formaldehyde adhesives for plywood

4.2.1.1.1 Cold and hot curing adhesives

Typical UF adhesive formulations for plywood are given in Table 4.

Table 4

Ingredients	Cold press	Hot press			
	CWR	CWR (1)	CWR (2)	WWR	BWR
UF resin powder	100	100	100	100	100
Water	200	200	140	125	100
Extender	50	80	35	25	15
Catalyst	5	3	2	2	2
Buffer	0-0.5	-	1	1	-
Fortifier	-	-	-	-	30

The formulations and proportions of ingredients given in Table 4 may require modification to suit different resin samples, extenders and fortifiers. Some examples of the preparation of UF resin adhesives for each grade of plywood using different extenders are briefly described below. The spread obtained with the adhesives will depend on species, veneer thickness and quality. Thicker veneers require a heavier spread. Press temperature, press time and pressure for manufacturing plywood should be carefully worked out for each situation.

4.2.1.1.2 Cold press CWR adhesive, groundnut meal extender

12.5 kg finely ground (-150 mesh) defatted groundnut meal is stirred into 50 kg water in a glue mixer with continuous stirring. 60 grams sodium hydroxide dissolved in one kg water is added and stirring continued for 15 minutes. 25 kg UF resin powder is added to the dispersion of groundnut meal and mixing continued

till a homogeneous dispersion is obtained. Finally 1.25 kg ammonium chloride dissolved in 2 kg water is added and mixed for about 10 minutes. The glue is then ready for use.

If instead of UF resin powder UF resin syrup of, say, 50 per cent concentration is used the quantity of water in which groundnut meal is dispersed is reduced to 25 kg and 50 kg resin syrup is used in the above adhesive mix.

The adhesive mix has a pot life of 3 - 4 hours at 30°C. It may be spread at the rate of 0.5 kg of adhesive mix per sq. metre (7.3 sq. metres/kg of solid UF resin). The veneer assembly should be pressed within 30 minutes after applying the adhesive on the first veneer. A clamping period of 8 to 16 hours is required at room temperature.

4.2.1.1.3 Hot press CWR adhesive, groundnut meal extender

10 kg defatted groundnut meal (-150 mesh) is soaked in 40 kg water for two hours. 0.58 kg liquor ammonia (sp. gr. 0.91) is added and mixed for about 10 minutes. 10 kg UF resin powder is then added and mixed to a smooth consistency followed by the addition of 0.2 kg ammonium chloride dissolved in 1 kg of water. The mixture is stirred for 15 minutes. 1.2 kg formalin (37%) is added and the stirring continued for another 10 minutes. The adhesive is then ready for use.

The pot life of the adhesive mix is about 4 hours at 30°C. It can be spread at the rate of 0.5 kg adhesive per sq. metre

(12 sq. metres/kg of solid UF). The assembly is then hot pressed at 105 to 110°C for 6 minutes for 4 mm thickness at 10 kg/cm². Veneer assembly should be hot pressed within 30 minutes after applying the adhesive.

4.2.1.1.4 Hot press CWR adhesive, wheat flour extender

20 kg finely powdered (-100 mesh) wheat flour is stirred into 50 kg water and mixed for 10 to 15 minutes. To this dispersion 25 kg UF resin powder is added with continuous stirring. After another 10 to 15 minutes of stirring, 0.75 kg of oxalic acid dissolved in 2 kg of water is added and mixed for 10 minutes. The adhesive mix is now ready for use.

The adhesive has a pot life of ^{about} 4 hours at 30°C. It may be spread at the rate of 0.45 kg adhesive mix per sq. metre (8.7 sq. metres/kg of solid ^{UF} resin). The veneer assembly should be pressed within one hour after applying the adhesive, at 10 kg/cm², and 105 - 110°C, for 4 - 5 minutes, for 4 mm thickness.

4.2.1.1.5 Hot press WWR adhesives

(a) Sal seed meal extender. 6.25 kg finely ground (-100 mesh) defatted sal seed meal is stirred into 30 kg of water. 0.1 kg liquor ammonia (0.91 sp. gr.) is added and mixed for about 15 minutes followed by 25 kg urea formaldehyde resin powder. 0.5 kg ammonium chloride dissolved in 2 kg water is added to the dispersion with continuous stirring. The adhesive mix is then ready for use.

The adhesive has a pot life of about 4 hours at 30°C. It may be spread at the rate of 0.4 kg adhesive mix per sq. metre (6.7 sq. metre/kg solid UF resin). The veneer assembly should be pressed within one hour after applying the adhesive, at 105 - 110°C for 4 to 5 minutes for 4 mm thick plywood at 10 kg/cm².

(b) Groundnut meal extender. 6.25 kg finely ground (-150 mesh) defatted groundnut meal is stirred into 30 kg water and mixed with 50 grams sodium hydroxide dissolved in 1 kg water. The mixture is allowed to stand for 15 minutes. 25 kg UF resin powder is added to the mixture with continuous stirring followed by 0.5 kg ammonium chloride dissolved in 2 kg water. Mixing is continued for 15 minutes. The adhesive mix is then ready for use.

The adhesive has a pot life of ^{about} 4 hours at 30°C. It may be spread at the rate of 0.4 kg adhesive mix per sq. metre (6.7 sq. metre/kg solid UF resin) on 1.5 mm veneer, 0.45 kg/sq. metre on 2.4 mm veneer, and 0.50 kg/sq. metre on 3.2 mm veneer. The veneer assembly should be pressed within one hour after applying the adhesive, at 10 kg/cm² and 105 - 110°C for 4 to 5 minutes for 4 mm thick plywood.

4.2.1.1.6 Hot press BWR adhesive, melamine resin fortified

25 kg UF resin powder is dispersed in 25 kg water. 3.75 kg china clay is added and the mixing continued. 7.5 kg melamine formaldehyde resin powder is added and stirred for about 15 minutes followed by 0.5 kg ammonium chloride dissolved in 2 kg water. Ten minutes more mixing and the adhesive is ready for use.

The adhesive has a pot life of 3 - 4 hours at 30°C. It may be spread at the rate of 0.5 kg adhesive mix per sq. metre (5.1 sq. metre/kg solid UF resin). The veneer assembly should be pressed within 2 hours after applying the adhesive, at 10 kg/cm², and 115 - 120°C for 5 - 6 minutes for 4 mm thick plywood.

4.2.1.2 Phenol formaldehyde resin adhesives for plywood

4.2.1.2.1 BWR adhesive, groundnut meal extender

5 kg defatted groundnut meal (-80 to -100 mesh) is dispersed by soaking in 25 kg water containing 0.25 kg sodium hydroxide for about 20 to 30 minutes. 25 kg liquid PF resin of 50 per cent solids is added and mixed for 15 minutes. The adhesive is then ready for use.

The adhesive has a pot life of more than 24 hours at 30°C. It may be spread at the rate of 0.48 kg adhesive per sq. metre (4.5 sq. metre/kg of the liquid PF resin). The assembly is pressed at 14 kg/cm², and 140-150°C for 8 minutes for 4 mm thick plywood, immediately after application of glue on veneer or up to 24 hours later.

For extension with proteinous materials and tannins, PF resin of relatively lower viscosity and greater water tolerance is used. Resin of such characteristics is prepared as in section 2.2 using a molar ratio of phenol to formaldehyde of 1 : 2.2. Five per cent sodium hydroxide on phenol is used as catalyst. The reaction is carried out at 90 - 95°C to get the desired degree of polymerization which is determined by viscosity (60 - 80 cp) and water tolerance (1 : 12). The resin is cooled immediately to room temperature by circulating cold water.

4.2.1.2.2 BWR adhesive, casein extender

10 kg casein is soaked in 40 kg water for 2 hours. 1.5 kg of a 10 per cent solution of borax is added with stirring which is continued. After 15 minutes 0.5 kg of 30 per cent formalin diluted with 4 kg water is added slowly. Finally 25 kg liquid PF resin (section 4.2.1.2.1) of 50 per cent solids is added and the mixing continued for 15 minutes. The adhesive is then ready for use.

The adhesive has a pot life of more than 8 hours at 30°C. It may be spread at the rate of 0.5 kg adhesive per sq. metre (6.4 sq. metre/kg of liquid PF resin). The glued veneers are assembled and pressed within 2 hours at 140 - 150°C for 8 minutes, for 4 mm thick plywood at 14 kg/cm².

4.2.1.2.3 BWP adhesive, coconut shell flour filler

10 kg liquid PF resin (50% solids, section 2.2.1) is mixed with 4.0 kg of coconut shell flour (-200 mesh) for about 15 minutes. The adhesive is then ready for use. It may be spread at the rate of 0.35 kg adhesive per sq. metre (3 sq. metre/kg of liquid PF resin). The glued veneers are assembled after 4 to 24 hours and pressed at 14 kg/cm², and 140 - 150°C for 8 minutes for 4 mm thick plywood.

4.2.1.2.4 BWP adhesive, mimosa tannin extender

5 kg finely ground (-150 mesh) defatted groundnut meal is stirred into 25 kg water. 0.5 kg trisodium phosphate dissolved in one kg water is added and mixed thoroughly. 25 kg mimosa/wattle tannin powder is added to the dispersion and stirring continued till a

homogeneous mixture is obtained. 12.5 kg liquid PF (section 4.2.1.2.1) is added and mixing continued for 15 minutes. Finally 5 kg formalin (37%) is added and stirred well. The adhesive is then ready for use.

The adhesive has a pH of 6.6 to 6.8 and a pot life of 4 - 6 hours at 30°C. It may be spread at the rate of 0.45 to 0.50 kg of adhesive per sq. metre. The veneer assembly may be prepressed immediately at 8 - 10 kg/cm² for 10 minutes followed by hot pressing within 2 hours at 14 kg/cm², and 120-130°C for 6 minutes for 4 mm thickness.

The adhesive mix has a strong smell of formalin. By using 2 kg paraformaldehyde instead of formalin and 1.0 kg trisodium phosphate this smell can be avoided. The adhesive mix has a pH of 7.2-7.5.

4.2.1.2.5 BWP adhesive, modified with quebracho tannin

6 kg chalk powder and 7 kg coconut shell flour are mixed with 100 kg liquid PF resin (section 2.21). 6 kg quebracho tannin extract dissolved in 7.5 kg water is added and stirred for 15 minutes. 0.2 kg paraformaldehyde or 1 kg formalin (37%) is added and mixed for 15 minutes. The adhesive mix is allowed to stand for one hour when it is ready for use.

The adhesive has a pot life of over 24 hours and should be spread at the rate of 0.32 kg adhesive per sq. metre (3.3 sq. metre/kg of liquid PF). The assembly is prepressed at 5 kg/cm² for 15 minutes followed by hot pressing at 115 - 125°C for 6 minutes at 14 - 19 kg/cm² depending on species.

4.2.2 Formaldehyde adhesives for particle board

4.2.2.1 Urea formaldehyde resin adhesives for particle board

75 kg UF liquid resin (66% solids) (section 2.1.2) is mixed with 25 kg water in a glue mixer. One kg ammonium chloride dissolved in 2 kg water is added and stirred for 15 minutes. Additives such as wax emulsion and preservatives may be added as required. The adhesive mix is now ready for use.

Pot life of the adhesive is about 4 hours at 30°C.

If a powder resin is used it has to be reconstituted with an equal weight of water.

4.2.2.2 Phenol formaldehyde resin adhesive for particle board

PF resin prepared as in section 2.2.1 is suitable for particle board manufacture. Wax emulsion, preservatives and other additives may be added as required.

4.2.3 Formaldehyde adhesives for assembly bonding

4.2.3.1 Urea formaldehyde gap-filling adhesive for assembly bonding

Gap-filling UF resin adhesives are often required where irregular surfaces or the lack of adequate pressure can leave gaps wider than 0.125 mm between the mating wood surfaces, for example in assembly gluing of wood for interior locations. Gap-filling property can be imparted to UF resin adhesive by the use of special additives such as benzyl alcohol, furfuryl alcohol and furfuraldehyde.

To one kg UF resin syrup of 66 per cent solids (section 2.1.1) 0.135 kg furfuraldehyde mixed with 0.135 kg wood flour is added and mixed. 20 g ammonium chloride dissolved in a little water and 10 g tricalcium phosphate are added and mixed thoroughly to obtain an adhesive ready for use.

4.2.3.2 Phenol formaldehyde resin adhesive for assembly bonding

10 kg liquid PF resin (section 4.2.1.2.1) is taken in a glue mixer and one kg wood flour (-200 mesh) is added with continuous stirring. 2 kg p-toluene sulphonic acid is added and mixing continued for 10 minutes. pH of the adhesive mix is 0.8 to 1.0. The pot life of the adhesive is around 2 hours at 25°C.

Wood joints being glued should be assembled within 30 minutes after applying the adhesive. A clamping period of 16 to 20 hours is required at 27 - 30°C.

4.2.3.3 Resorcinol formaldehyde and phenol resorcinol formaldehyde resin adhesives for assembly bonding

One kg RF or PRF is mixed with 100 grams wood flour (-200 mesh). 150 grams finely powdered paraformaldehyde is added and mixed to obtain a homogeneous mixture which is then ready for use. The pot life of the adhesive is about 2 hours at 25°C. It is desirable to keep the adhesive cooled to about 20-25°C in a cold water bath to prolong the pot life.

The joints being glued should be assembled within 30 minutes after applying the adhesive. A clamping period of 16 to 20 hours is required at about 30°C.

4.3 Polyvinyl acetate resin adhesive

4.3.1 Polyvinyl acetate resin adhesive for wood working

Polyvinyl acetate resin emulsion of 55 per cent concentration is mixed with the ingredients mentioned below to get a homogeneous dispersion. The adhesive is then ready for use.

Water	..	50.0 kg
Polyvinyl alcohol (fully hydrolysed)	..	5.0 kg
China clay or chalk powder	..	10.0 kg
PVAc emulsion	..	31.0 kg
Dibutyl phthalate	..	3.5 kg
Preservative	..	0.3 kg
Defoamer	..	0.2 kg

The adhesive has a pot life of over one year.

5 Properties of Synthetic Resin Adhesives

5.1 Table of properties of synthetic resin adhesives

Adhesive	Form	Properties	Preparation and application	Typical uses for wood bonding
Urea formaldehyde resin	Powder or liquid (45% to 66% solids)	High dry strength, low resistance to moisture and damp conditions. Light colour. Relatively low cost, poor resistance to crazing. Storage life: powder 6 to 18 months, liquid resin 1-6 months.	Powder mixed with water at room temperature. Can be cured above 20°C but for rapid curing at room temperature acid catalyst is required. Mostly hot press gluing is practised at 105°C-110°C. Resin can be extended with starch and proteinous materials.	Plywood for interior use and furniture; interior particle board; interior flushdoors; for splicing veneers.
Melamine formaldehyde resin	Usually marketed as a powder with or without catalyst	High in both wet and dry strength; very resistant to moisture and damp conditions. High cost. Colourless. Storage life about one year for powder.	Powder mixed with water at room temperature. Hot press (115°C-125°C) and intermediate temperature (70°C-90°C) setting.	Primarily as fortifier for urea resins for plywood; end jointing and edge gluing of lumber; veneer splicing
Phenol formaldehyde resin	Liquid, powder, and dry film. Most commonly sold as aqueous alkaline dispersions.	High in both wet and dry strength; very resistant to moisture and damp conditions; moderate chemical resistance. Rather high cost. Imparts dark red colour. Resistant to crazing. Storage life: film and powder 3-6 months, liquid 1-3 months.	Film form used as received; powder form mixed with solvent, often alcohol and water at room temperature; liquid form used with fillers. High temperature setting (125-150°C) and as modified resin which is acid cured at intermediate temperature (25-95°C). Can be extended with starch, proteinous and tannin materials.	Semi-exterior and exterior plywood; moisture resistant particle board.

Adhesive	Form	Properties	Preparation and application	Typical uses for wood bonding
Resorcinol formaldehyde resin and phenol resorcinol formaldehyde resin	Available in liquid form; hardeners supplied separately.	High in both wet and dry strength; very resistant to moisture and damp conditions. Moderately resistant to chemicals. Imparts dark red colour. High cost. Storage life 2 months for PRF and 1 year for RF.	Mixed with hardener and applied at room temperatures; cures at room temperature on most species.	Primarily for laminated timbers and building components of wood, plywood and panels.
Epoxy resins	Viscous liquids	Completely reactive, good adhesion to metals, glass, certain plastics and wood products. Storage life one year. Resistant to crazing. No by-product is formed on curing, does not shrink on hardening.	Available in two parts, resin and curing agent, both liquid; mixed at the point of use. Applied at room temperature depending on the formulations.	Composite joints of metal and other materials to wood.
Polyvinyl acetate	Viscous liquid	Generally high in dry strength; low resistance to moisture and elevated temperatures; Joints tend to yield under continued stresses. White in colour. Storage life more than an year.	Readily usable, applied and pressed at room temperature. Sets immediately upon loss of solvent. Thermoplastic.	Furniture assembly, joinery, plastic laminates.

5.2 Technical limitations

The manufacture and use of synthetic resins and adhesives are skilled operations requiring trained personnel. Close control is required in manufacture to ensure that products of the desired physical and chemical properties are obtained. The quality of the raw materials have to be checked and maintained. For example, formalin for UF resin should be free from excess iron and formic acid. Urea should be free from excess of acid forming salts.

The moisture resistance and durability of UF resin bonds are not of a sufficiently high order for tropical environments. UF bonded plywood and particle board show loss of strength in environments of high temperature and fluctuating humidity. The possibility of wetting of panels by rain water even in interior locations has to be borne in mind.

In the manufacture of PF resins temperature control is very important. Storage life of the resin will be affected in the absence of proper control. There is also the danger of the resin gelling in the kettle if the exothermic reaction is not controlled.

The dark colour of PF resin is a limitation in the manufacture of wood based panels with thin face veneers, particularly decorative veneers. Though melamine fortified UF resin adhesive gives a colourless bond of high durability, there is need for a better bond for tropical environments. In this context it may be pointed out that in most developing countries furniture and other items manufactured from wood and wood-based panels would be expected to

have a very long life. Much work also remains to be done on the gluing characteristics of different timbers with PF resin adhesives.

RF and PRF adhesives are both very expensive. Gluing characteristics of timbers and wood-based panels, especially after preservative treatment, with these adhesives have to be determined.

The main drawbacks to the use of epoxy resins are their high cost and the possibility of some operators getting dermatitis by handling amine hardeners.

Polyvinyl acetate resin adhesives are sensitive to moisture and show creep under sustained load.

Close control of quality of fillers and extenders for adhesives has to be maintained. Most of these materials being of natural origin are likely to show variation in quality and deterioration by microorganisms. Seed meal extenders should be solvent extracted quality to ensure low fat content.

In cost oriented production there is danger of the excessive use of extenders. Low viscosity resin may even be specially made for this purpose. This has to be guarded against.

Finally, the quality and performance of an adhesive bond does not depend upon the adhesive alone. Preparation and characteristics of surfaces to be glued require as much care and attention as for the preparation and application of the adhesive.

6 Economics of Synthetic Resin Adhesives

6.1 Economy of scale

The minimum economic size for a synthetic resin plant is considered to be 20,000 tonnes per annum or more.

This scale of operation can be considered in a developing country only as an export-oriented industry. Economics of scale are significant and have to be considered in such a large-scale industry. On the other hand market prospects in developing countries suggest small-scale operations to meet local demand. When wood-based panels become known and the phase of initial end-use penetration is over the industry will grow and the local markets will absorb increasing quantities of panel materials. Small scale operations have the advantage of lower requirements of capital and investment. It may also be possible to fabricate resin making equipment locally. Local production of resins may not however be competitive without trade regulations or subsidies from the Government.

A disadvantage of local production of resin is the loss of some of the non-economic advantages, such as research and development and technical service, in buying from a resin manufacturer. Cooperative action by several small-scale manufacturers of resin is a possible way of overcoming these disadvantages.

Resin manufacturing units in India of 600 to 7000 tonnee annual capacity supply resin to wood-based panel manufacturere whose annual requirement is 30 to 600 tonnes. Several wood-based panel mills produce 150 to 1500 tonnes resins per year for use in their own mille. In these mills the technical etaff responsible

for resin production are also responsible for its use in the wood-based panel plant. The smallest resin manufacturing plant in operation has a capacity of 250 kg resin per charge. All these resin manufacturing units are viable so long as the demand for panel materials exist. A substantial portion of the panels manufactured is now exported.

6.2 Import costs and cost of local production

The cost of synthetic resins in the international market may be expected to be much less than the cost of similar resins produced on a small scale in a developing country. The present import cost of urea formaldehyde resin powder is \$ 416 to \$ 510 per tonne CIF Indian ports. One of the largest Indian manufacturers offers UF resin powder at Rs.8000 or \$ 727 per tonne. Taking the cost of urea as Rs.2000 per tonne, and 37% formalin as Rs.3000 per tonne, cost of 50 per cent UF resin syrup produced in a 2 tonne capacity kettle in a plywood plant in India is estimated to be Rs.3200 or \$ 290. The corresponding cost of liquid phenol formaldehyde resin (50%) is estimated to be Rs.7400 or \$ 673 for a phenol cost of Rs.12600 per tonne. Local production costs are subject to very wide fluctuations. Cost figures given above do not include local taxes and duties which are quite substantial.

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8. Glossary

1. Bubble viscometer : A device to determine the viscosity of a liquid by measuring the speed with which a bubble of air rises in the liquid.
2. CP : Centi-poise; one-hundredth of a poise (gm/cm, sec). It is a quantitative measure of the viscosity (or consistency).
3. Flow cup : A practical and easy device for determining the viscosity of a liquid. Viscosities are determined by the time in seconds taken by the liquid to effuse from an orifice of standard dimensions.
4. pH : A measure of acidity or alkalinity on a scale from 1 to 14. The logarithm (to base 10) of the reciprocal of the hydrogen ion concentration in an aqueous solution determines the numerical value. Water is neutral at a pH of 7.
5. Pot life : The time between the mixing of the constituent parts of an adhesive and its reaching a stage when it is no longer usable.
6. Storage life (shelf life) : The period for which the adhesive or adhesive components can be stored without affecting their suitability for use in accordance with the standard.
7. Viscometer : A device for measuring viscosity.
8. Viscosity : A parameter which defines the resistance of a liquid to shear forces. The higher the viscosity, greater is the force required to make it flow.
9. Water tolerance : The parts by volume of water which is compatible with a known volume of resin during dispersion as indicated by the appearance of turbidity.

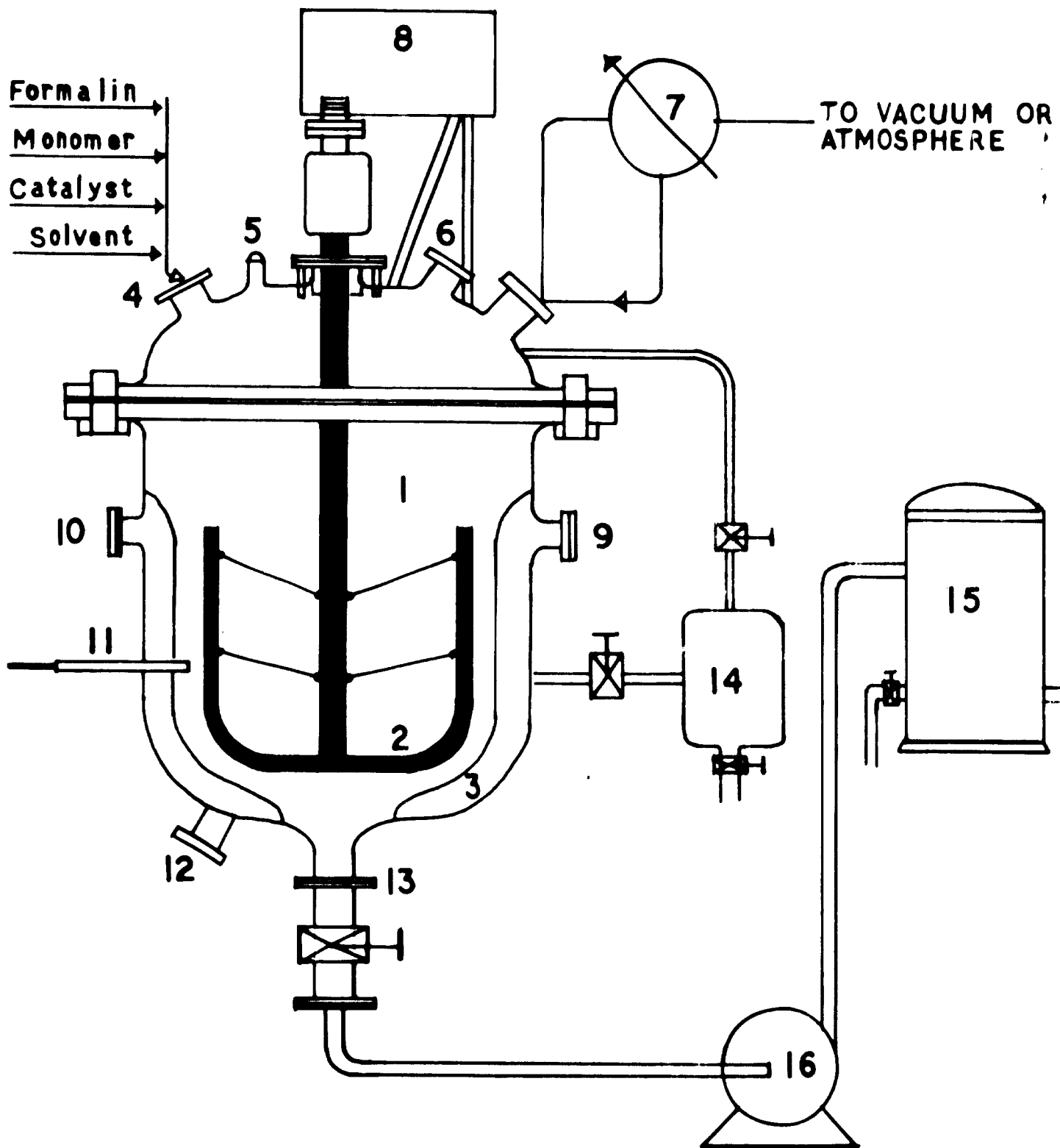
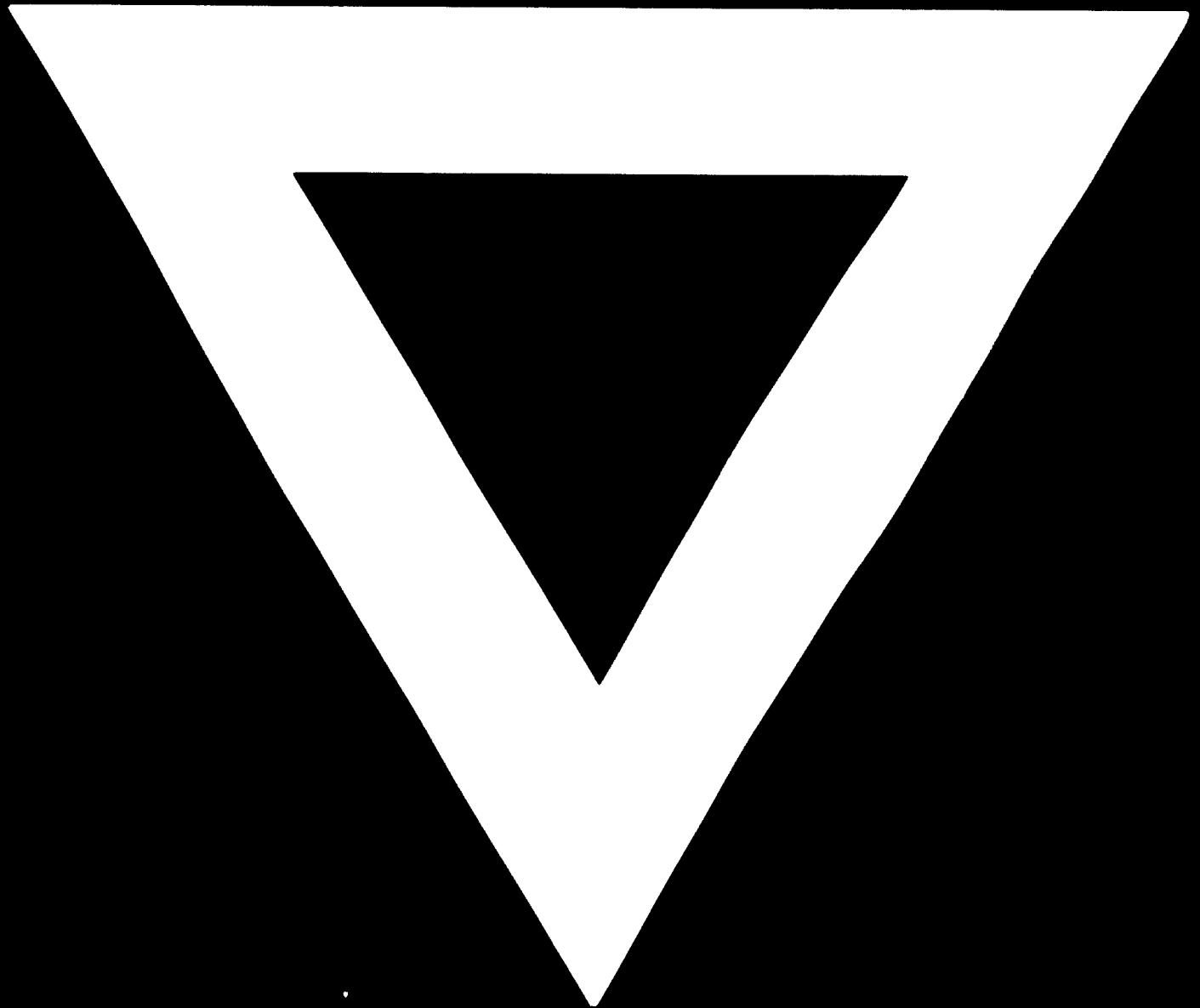


Fig.1. SCHEMATIC DIAGRAM OF A RESIN MANUFACTURING UNIT



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