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TEFLON* TFE-FLUOROCARBON RESIN DISPERSIONS PROPERTIES AND PROCESSING TECHNIQUES

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2. COATED GLASS FABRICS AND YARNS for electrical insulation in motors, generators, and transformers; wrapped insulation on wire for high-temperature applications; non-adhesive separator sheets for laminating, and press blankets; gaskets, and rigid high-temperature laminates.
3. IMPREGNATED ASBESTOS OR TFE FIBER AND BRAID for gasket and packing uses.
4. SURFACE COATINGS for non-adhesive and low friction uses².
5. WIRE ENAMELS³ for magnet wire in small relays and transformers.
6. BLENDS with other resins and fillers, including filled molding powders for molding and extrusion.
7. TEXTILE FINISHES for yarns and fabrics.

"Teflon" TFE-fluorocarbon aqueous dispersions are milky white liquids consisting of very small particles of tetrafluoroethylene resin suspended in water. The high utility of these dispersions is due to their fluid form. This property is especially useful in the case of "Teflon" TFE-fluorocarbon resins, because these resins are not suitable for processing in molten or dissolved form.

USES

Uses for TFE dispersions fall into the general categories of coating, impregnation, finishing, and blending. More specifically the material is used for the following:

1. CAST FILM for capacitor layer insulation and diaphragms.

*Reg. U. S. Patent Office

PROPERTIES OF "TEFLON" TFE-FLUOROCARBON DISPERSIONS

TFE dispersions are hydrophobic, negatively charged colloids containing particles of 0.05 to 0.5 micron diameter suspended in water. Pertinent properties are as follows:

pH—

As normally supplied, the pH is about 10. If necessary, the pH can be reduced by addition of either inorganic or organic acids; the latter are

¹"TEFLON" TFE-Fluorocarbon Fibers are available from the Textile Fibers Department, E. I. du Pont de Nemours & Co. (Inc.), Wilmington, Delaware.

²Finishes and wire enamels based on "TEFLON" tetrafluoroethylene resins manufactured by Fabrics & Finishes Department, E. I. du Pont de Nemours & Co. (Inc.), Wilmington, Delaware.

much preferred. Care must be taken to avoid adding excessive quantities of acid, because high ion concentrations will cause coagulation.

Stability—

The dispersion will settle somewhat on prolonged standing or on prolonged heating above 160°F. but can generally be redispersed by mild agitation. Stocks being stored for an indefinite period should be redispersed at least once a month by tumbling or rolling in the container. The dispersion should be protected at all times against freezing, which will cause an irreversible coagulation. High-speed stirring and addition of electrolytes or water-miscible solvents will also usually lead to irreversible coagulation.

Specific Gravity—

The relationship between concentration and specific gravity at 77°F. is given in the following table, along with useful equivalents:

Per Cent Solids	Specific Gravity	Grams solids per cc.	Pounds of solids per gallon
35	1.24	.436	3.72
40	1.29	.515	4.30
45	1.34	.601	5.01
50	1.38	.693	5.79
60	1.50	.900	7.50

Critical Thickness—

The critical thickness, or maximum thickness of film that can be made in a single casting without formation of cracks when the deposited polymer is dried, is between 0.5- and 1.5-mils depending on the application conditions and the shape being coated. Thus, coatings no thicker than 0.5- to 1.5-mils should be deposited in a single cycle in order to avoid forming such cracks that cannot be fused together in baking. Thicker coatings are made by multiple dipping and baking operations.

"Teflon" 30 and "Teflon" 30B contain approximately 60 percent resin and 6 percent "Triton" X-100, based on weight of solids. "Teflon" 42, a 35 percent solids dispersion, contains no "Triton", but is stabilized with an immiscible liquid. Further details on the characteristics of these resins may be found in the following Product Sheets: A-46473 ("Teflon" 30), A-46474 ("Teflon" 30B) and A-45012 ("Teflon" 42).

FORMULATIONS

"Teflon" TFE-fluorocarbon dispersants as furnished contain "Triton" X-100*, a non-ionic wetting agent which has good wetting properties and a

*Rohm and Haas, Bristol, Pennsylvania

minimum tendency to foam. Moreover, it can be decomposed cleanly at the temperatures required for baking, leaving a contaminant-free polymer.

In many cases, it will be desirable to supplement the formulation to increase the viscosity, to improve the penetration, or to increase the critical thickness of TFE dispersions.

The viscosity may also be adjusted by adding water soluble thickeners such as Carbopol 934 (B. F. Goodrich Co.). As little as 0.1% of this thickener will increase the viscosity of a 60% solids dispersion to 600 centipoises. Non-ionic and anionic types of dispersants are acceptable. The non-ionic wetting agents are generally preferred because they are less likely, in low concentrations, to induce abnormal viscosities resulting from thixotropic behavior. Too, they can be burned out most readily at polymer-sintering temperatures without leaving inorganic residues in the product. Dispersing agents of the cationic type are normally unsatisfactory because they will flocculate the dispersion.

It is important to note that only mild agitation should be used in adding any material to the dispersion. High-speed stirring should be avoided, because it will lead to coagulation.

CASTING AND COATING

The availability of "Teflon" TFE-resins in the form of aqueous dispersions makes it possible to cast films and to apply enamel-type coatings of the material. In addition, spray finishes based on dispersions of "Teflon", and information on their uses are available from the Du Pont Fabrics and Finishes Department.

In the case of film, "Teflon" TFE dispersion is applied by dipping or flowing onto the surface, followed by drying to remove water (or other media, in the case of some finishes), and fusing the dried polymer at 675° to 750°F. Dip-coating has proved preferable in most cases because of its adaptability to continuous application of a uniform coating. A dispersion concentration of 45 to 50% solids by weight and 6 to 9% (on solids basis) wetting agent are recommended for optimum wetting characteristics. The thickness of dispersion layer picked up during each dip should be limited to about 0.5-mil in order to avoid the formation of cracks upon drying as well as to permit reasonably rapid vaporization of the dispersing agent during the sintering operation. Thicker films can be applied in a single pass if a high dielectric strength film is not needed. The amount of pick-up is influenced by the viscosity of the dispersion, the coating speed, and the roughness of the surface being coated, but it can be controlled by means of a doctor knife, as shown in Figure 1. As many coats may be applied as desired. It is not unusual for a coat to be sintered before another one is applied.

After dipping, the next step is removal of the water from the deposited dispersion. This may be done under infra-red lamps, or in a forced-convection air-oven. In either case, the speed of water removal will be limited by the tendency for bubble formation. For a 1-mil film, infra-red drying will take about 15 seconds. The air-oven technique will take longer—sometimes as long as 1 to 2 minutes at 185° to 205°F.

Drying is followed by a baking and sintering operation, during which two things occur—the wetting agent is volatilized, and the discrete solid resin particles are sintered into a homogeneous film. Both operations can be carried out simultaneously at the sintering temperature, however, it is recommended that the baking step be conducted separately at a lower temperature to minimize the decomposition of the wetting agent to a carbonaceous residue. Baking or volatilization of the wetting agent is carried out in an air oven maintained at a temperature of about 375°F.; temperatures of 300° to 425°F. are most commonly used, depending on the equipment and desired baking time.

Sintering or fusion of the resin particles takes place almost instantaneously on reaching temperatures of 675° to 750°F. Most of the wetting agent is volatilized at the baking temperature, but a small amount remains and decomposes during sintering, leaving a minute carbonaceous residue that can be removed by oxidation if the sintering time is extended. Whether or not this residue should be baked out depends on the particular application; it causes discoloration, detracts from the electrical properties, and makes film stripping very difficult.

If none of these effects are important factors, the sintering cycle can be relatively short—about 3 minutes for a 1-mil film. However, for complete removal of stabilizer residues, a sintering cycle of about 8 to 10 minutes is preferred for a 1-mil film. The cycle increases to about 30 minutes for a 2-mil film. Hence, multiple applications of one mil each are more desirable from the standpoint of sintering time, even if the critical thickness were great enough to permit thicker deposits in a single cycle. Multiple dips, with baking and sintering between dips, result in good homogeneity.

When unsupported film is made by casting, the final step is stripping of the film from the metal surface. Rapid cooling of the film from the sintering temperature by quenching in cold air or water promotes ease of stripping. By the same token, slow cooling and even annealing promote good adhesion. Other factors that facilitate stripping are complete removal of wetting agent and maintenance of a metallic surface free of corrosion and in a fairly high state of polish. When the same surface is used repeatedly, corrosion at the high temperatures employed in the sintering oven becomes a problem. Ordinary steel and even stainless steel corrode rapidly, while nickel, chromium and silver are more resistant. Nickel appears to give the best results. Washing the surface in dilute acid and buffing after stripping each film are helpful.

The procedure outlined above is based on limited study of the variables involved in casting films from TFE dispersions. Undoubtedly, modifications to meet specific needs can be made to advantage. Typical properties of thin film cast from TFE dispersions in this manner are given in Table 1.

TABLE I—PROPERTIES OF THIN FILMS CAST FROM "TEFLON" TFE-FLUOROCARBON

Properties	Units	A.S.T.M. Method	Value
Tensile strength, 77°F.	Psi	D882	3,000-4,000
Elongation, 77°F.	%	D882	300-500
Tear strength, Die C	lb./in.	D624	400-800
Stiffness, 77°F.	Psi	D747	40,000-90,000
Moisture-permeability, (0.001 inch thickness)	gm./100 sq. in./day		0.2
Dielectric constant, 1000 cycles		D150	2.0-2.2
Power factor, 1000 cycles		D150	<0.0003
Volume resistivity	ohm-cm.	D257	>10 ¹⁵
Dielectric strength, (short time, 0.002 inch)	v/mil	D149	<3,000
Electrical flaws at 800 v/mil (0.001 to 0.004 inch films)	flaws/100 ft. / ½-in. width	D1389 modified	0-8
Specific heat, 100° to 260°F.	Btu./lb./°F.		0.25
Water absorption	%	D570	<0.1
Brittleness temperature	°F.		<-100
Specific gravity	—	D792	2.15-2.20

IMPREGNATION

A variety of porous structures can be impregnated with TFE dispersions by treatment with the dispersion. The dispersion is well suited for impregnation because of its low viscosity, extremely small particles, and the effect of the dispersing agent which aids in wetting the surfaces of interstices, promoting capillary action. After dipping and drying, the deposit of resin may or may not be sintered depending on the requirements of the application.

Sintered coatings on woven fabrics and mats of glass or asbestos fibers are prepared by much the same procedure as that described previously for cast films. It is, in fact, a matter of casting films on a discontinuous surface. Because of the critical thickness factor, multiple dips, with sintering between dips, are essential to build heavy coatings of good homogeneity.

Impregnated materials with a useful degree of chemical resistance and the non-adhesive property of "Teflon" can be prepared without sintering the deposited polymer. This involves merely dipping and drying. The wetting agent may be removed by

heating to about 375°F. The required time will be a matter of a few minutes. Pressure is usually applied to the impregnated material, either before or after drying, to aid in holding the "Teflon" resin in place. For example, a fabric or mat material can be calendered continuously as an integral part of the treatment, or the dried material can be compressed in a mold as an after-treatment. Thus, in addition to glass and asbestos, less heat-resistant fibers can also be treated.

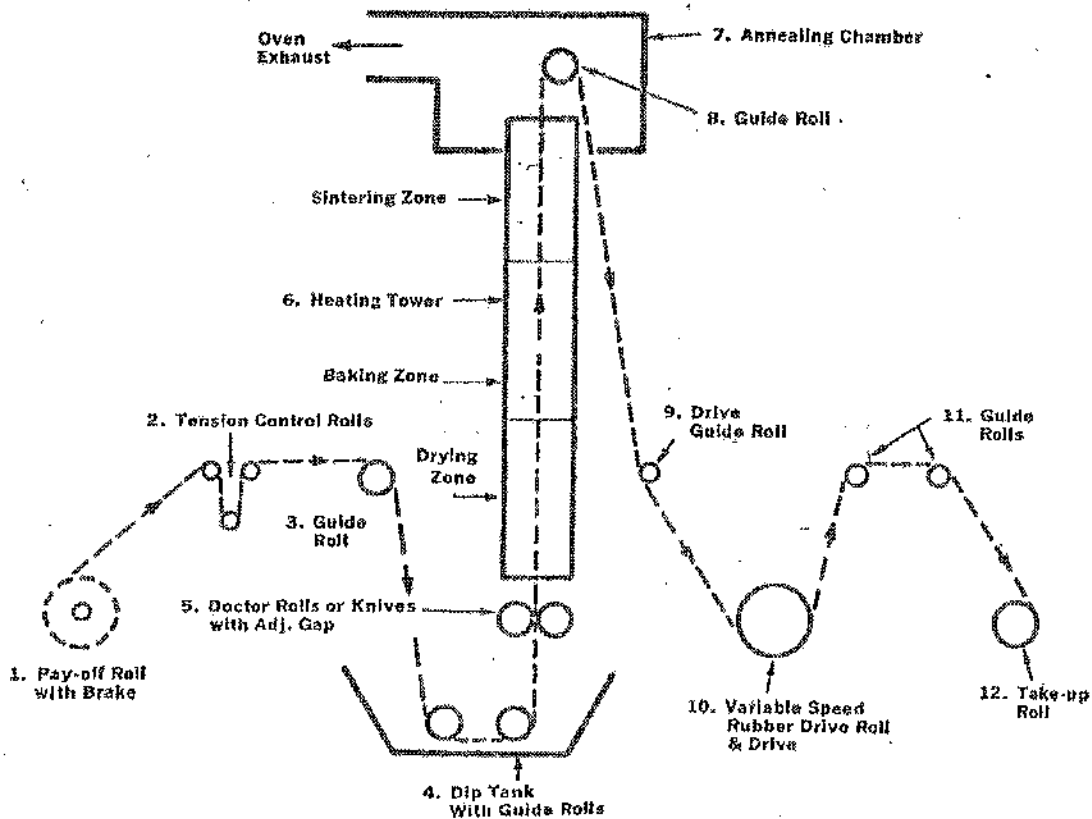
GLASS FABRIC COATING

Typical equipment for impregnating and coating glass cloth with "Teflon" dispersion is shown in Figure 1. It includes:

- (1) a pay-off roll for the cloth
- (2) a dip tank for the dispersion
- (3) a drying and sintering zone
- (4) a take-up roll.

The dip tank should be constructed of stainless steel. The tank should be equipped with at least one submerged rigid rod, preferably of "Teflon"

FIGURE 1
EQUIPMENT FOR IMPREGNATING GLASS FABRICS



for easy sliding of the glass cloth. If rotating rolls are used, the roll must be entirely submerged and sleeve bearings lubricated only by the liquid should be used.

Vertical towers capable of attaining 750°F. are used for drying, baking and sintering the coating. Although ovens with two heating stages can be employed, it is preferable to use a tower with three separately controlled zones. Ideally, the first (drying) zone should operate at 200°F. If a second zone is available, it may be operated at about 330° to 375°F. to remove the major portion of the wetting agent prior to sintering. Coatings should be sintered between 680° and 750°F. Higher temperatures may be used, but these will reduce the mechanical strength of the glass fabrics. It is recommended that a short annealing chamber be provided at the exit of the oven. This may take the form of an enclosure around the roll at the top of the oven. This chamber which prevents too rapid cooling of the coated fabric will reduce wetting problems when applying multiple coatings. The tower can be heated by gas, using vanes and recirculation of air, or by electrical heat with recirculated air. Air flow rates must not be excessive (a gentle breeze is needed) or fabric flutter may cause uneven coating. The tower should be wide enough to handle cloth 38 inches wide, since this is the standard width in which glass cloth is supplied. The third (sintering) zone should be about half the height of the tower. An overall height of 12 to 15 feet will permit production rates of 5 to 9 feet per minute.

Ventilating ducts and an exhaust blower to remove volatile products and traces of decomposition products from the ovens should be used (see Safe Handling).

Glass greige goods that contain approximately 2% of a starch-oil size, are most commonly used for impregnation. In special cases, other finishes in the glass may be employed.

Depending on the fabric weave and the thickness desired, two to four coats of the "Teflon" resin are usually required.

Bath compositions will also vary with the type of fabric and the coating speed. Ordinarily, the bath will contain 45 to 55% solids and 6 to 9% wetting agent (solids basis). For a 3-mil 108 glass fabric coated with the unit shown in Figure 1, the following conditions have proven satisfactory using both "Teflon" 30 and "Teflon" 30B:

Solids content—51%

Wetting agent—none added . . . 6% based on solids

Coating Speed—5 to 6 feet per minute

Two coats required.

LAMINATION

Standard laminating presses can be used for the production of laminates of glass cloth coated with "Teflon". Presses should be equipped with elec-

trically heated platens capable of heating the coated glass cloth to a temperature of 750°F. and have sufficient tonnage to apply 1,000 lbs./sq. in. The platens should be cored for cooling water, so that the laminates can be cooled fairly quickly, and the cycle time reduced.

Laminates of coated glass cloth are made by cutting the cloth to the desired size, stacking the cut pieces by hand, pressing the assembly between heated platens, and cooling. Before pressure is applied, it is important that the individual layers be flat and that there be no wrinkles. Pressure should be applied slowly so that air which will expand during heating and cause blisters will not be entrapped.

There is some tendency for the "Teflon" resin to stick to heated metal platens during lamination. To eliminate this, a foil of aluminum can be placed on both the top and bottom of the lay-up. Silicone mold releases are helpful. After removal of the laminate from the press, the foil can be stripped off easily.

It is important that the platens of the press be parallel, so that pressure will be applied equally to all sections of the laminate. Chrome-plated platens or press-polished plates are generally used to prevent rusting, and to impart a good surface to the finished laminate.

Normal laminating pressures and temperatures are 1,000 lb./sq. in. and 690° to 720°F., respectively. Although pressures are not critical, the pressure must be great enough to squeeze residual air out, and to force the individual layers of coated cloth into intimate contact. The heat must be applied at temperatures above 621°F. long enough to fuse all of the TFE resin.

BLENDS

Homogeneous mixtures of "Teflon" tetrafluoroethylene resins with a variety of materials can be prepared from the dispersion. Broadly, the procedure is simply a matter of mixing the material to be blended into the dispersion. Solid materials should be in a finely divided state, and best results will usually be obtained by suspending the solid in water before mixing with the dispersion. Systems in immiscible liquids can frequently be processed through use of mutually miscible solvents such as acetone or alcohol, though coagulation of the solid resin particles will occur immediately on mixing.

The preparation of filled molding powders by coagulating "Teflon" dispersion in the presence of a filler is described in Information Bulletin No. X-91C.

SAFE HANDLING PRACTICES

Research at the Haskell Laboratory for Toxicology and Industrial Medicine of the Du Pont Company and in university and government laboratories indicates that at processing temperatures "Teflon"

fluorocarbon resins give off volatile products which can affect laboratory animals if the exposure is long enough and the quantity of resin is great enough.

Animal feeding tests indicate that "Teflon" fluorocarbon resins have no observable detrimental effect when taken internally. Other animal tests similarly indicate that the resins do not produce skin irritation. These laboratory results are substantiated by the fact that there has been reported to us no case of skin irritation arising from the handling of "Teflon" resins at ordinary temperatures.

Direct experiments with animals have been supplemented, where practical, with measurements of weight loss rates and analysis of the products evolved on heating "Teflon" resins in air. The quantities of decomposition evolved at temperatures below 800°F. have been so small that complete identification of the evolved products has not been feasible with existing analytical techniques. However, at higher temperatures, 930° to 1020°F., sufficient amounts of decomposition products are given off for identification. At these temperatures the major pyrolysis product is the monomer tetrafluoroethylene, which is relatively non-toxic and markedly less hazardous in this respect than vapors of cleaning fluids such as carbon tetrachloride, benzene or gasoline. The minor but toxic part of the decomposition products consists of very small amounts of perfluorinated vapors varying in chain length from C₃ to C₆, a waxy sublimate and, if the pyrolysis is carried out in the presence of moisture, small amounts of hydrogen fluoride.

Industrial experience in the handling of "Teflon" resins is extensive. Large quantities of these resins have been processed safely by many different fabricators and in a variety of operations. The record clearly establishes that "Teflon" resins can be processed at elevated temperatures without

hazard if proper ventilation is used. However, if adequate ventilation is not provided and workers inhale the fumes of heated "Teflon" resins in sufficient quantities, influenza-like symptoms may follow. These symptoms do not ordinarily occur until several hours after exposure, and pass off within 36 to 48 hours, even in the absence of treatment. Observations indicate that these attacks do not have any lasting effects and that the effects are not cumulative. When such an attack does occur, it usually follows exposure to vapors evolved from the polymer at 545°F. or above without adequate ventilation, or from smoking tobacco or cigarettes contaminated with the polymer.

Although there is no record of workers being seriously injured by fumes from heated "Teflon" or its thermal decomposition products, fumes are increasingly toxic in heavy concentrations, just as are the fumes or decomposition products of many common resins, paints, elastomers and solvents, as well as naturally occurring polymeric materials like wood, silk, wool and rubber. Therefore, the ventilation precautions to be observed when heating "Teflon" are the same as those which should be observed in heating many types of conventional materials.

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