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Dispersion Techniques for Carbopol®* Polymers

Carbopol® polymers are highly water and polar solvent soluble polymers which function as efficient thickeners and stabilizers for suspensions or emulsions. Much of the exceptional utility of the Carbopol® polymers results from the hydrophilic nature of the polymer. A single particle of Carbopol® polymer will wet out very rapidly when put into water. Like many fine particulate powders, Carbopol® polymers tend to clump when improperly added to the solvent.

The surface of a powder aggregate solvates to form a tough outer layer which prevents complete wetting of the interior polymer particles. This results in dispersion defects such as grainy texture, reduced viscosity or the presence of insoluble particles resembling fish eyes.

To prepare high quality and reproducible dispersions of Carbopol® polymers, the proper dispersing technique must be carefully followed.

I. Direct Addition of Carbopol® Polymers into Water

The proper method to use when adding Carbopol® directly to water depends on the quantity and concentration of the dispersion to be prepared.

A. Small Quantity Batch Dispersions

Dispersions of several hundred milliliters to several gallons with concentrations of up to 1.5 weight percent Carbopol® polymer can be practically prepared by sifting the dry polymer into rapidly agitating water.

Aqueous surface wetting and dispersion of Carbopol® polymer is best achieved with moderate rate agitation of approximately 800-1200 rpm. Motorized mixers such as the Eppenbach, Colframo, Arde-Barinco, Janke and Kunkel or Lightnin' Mixer with a conventional open-blade impeller (pitched marine or saw tooth propeller) are most appropriate. Extremely high-shear mixers such as Waring blenders or rotor-stator homogenizers should be avoided or carefully employed. The mixing intensity generated by this type of mixer can shear the opened (hydrated) polymers resulting in permanent viscosity loss. In some cases, this loss can be as high as 50%. Conventional impellers, such as propellers or turbines do not impart excessively high shear rates. They can be used to mix mucilages for extended periods with virtually no decrease in polymer efficiency.

If in-line mixers such as colloid mills or homogenizers are used, the Carbopol® polymer should be initially wetted-out with moderate rate agitation. Therefore, the mixing time using the high shear mixers will be minimized. If a homogenizer is employed, strive to use the lowest shear rate and the shortest mixing time to achieve a homogeneous mixture.

When mixing, submerge the impeller until it is very close to the bottom of the vessel and angle the mixer to generate a vortex which is one to one-and-one half impeller diameters. Slowly sift the polymer into the vortex of the agitating liquid (approximately 1200 rpm). Continue the agitation for about 20 minutes or until a lump-free dispersion is attained. In some cases when a Lightnin' Mixer is employed, two impellers on a common shaft are recommended. The top impeller rapidly disperses the polymer before the undesirable, partially-hydrated lumps are formed. A sifting device such as a coarse sieve or a non-corrosive 20-mesh metallic screen should be used to slowly sprinkle the Carbopol® polymer into the vortex of the water generated by the mixer. The sifting method allows the slow, controlled addition of the dry Carbopol® polymer, enabling each particle to wet out on the water vortex. The sieve or screen permits the

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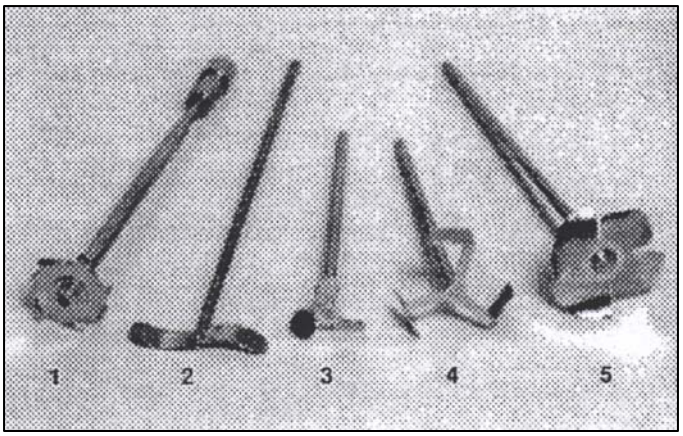
break-up of any loose polymer or agglomerates due to the static charge on the particles. A metallic screen decreases the tendency to build more static charge on the particles as they are sifted.

Once all the dry polymer has been introduced, continue agitating at the moderate to rapid rate for 10-15 minutes. Reduce the speed to avoid the entrapment of excess air.

For emulsions, add oils and emulsifiers to form the emulsion prior to neutralizing the Carbopol® to build emulsion viscosity. Neutralization of Carbopol® is accomplished with a suitable alkali or amine base. For the order of neutralizer addition in gel products, see the Carbopol® Troubleshooting Guide.



1. Cowles Blade—Not recommended due to high shear.
2. Paddle Blade—Frequently used when blending operations follow initial dispersion of Carbopol polymer.
3. 3-Blade Marine Impeller—Excellent choice for dispersing Carbopol® polymer. Impeller of choice when making emulsions.
4. Foddler Impeller—Poor mixing due to low shear.
5. Hi-Lift Impeller—High shear: use with caution.

Note: Dispersions of Carbopol® polymers made to a precise solids level for analytical viscosity testing should follow Lubrizol Standard Testing Procedure 430-I, "Brookfield Viscosity (low shear)."

Selecting Neutralizers for High Alcohol Solutions

- Metallic alkali (NaOH, KOH, etc.) will neutralize Carbopol® polymers with less than 20% ethanol.
- Triethanolamine (TEA) will neutralize Carbopol® polymers up to 50% ethanol solution.
- 2-Hydroxypropyl ethylene diamine (Quadrol®) will neutralize Carbopol® polymers up to 70% ethanol solution.

B. Large Quantity Batch Dispersions

Dispersion volumes ranging from tens of gallons to hundreds of gallons at concentrations of Carbopol® polymers of up to three weight percent may be prepared with an eductor or flocculant disperser. This technique is a fast, inexpensive means of dispersing Carbopol® polymers with reduced dusting.

Figure 1 shows how the rate of dispersion increases with an increase in water pressure. Figure 2 shows the basic design of the eductor. It is a small, inexpensive, metal or plastic, non-moving plumbing valve which exerts a partial vacuum on the powder inlet via the Bernoulli effect. Carbopol® polymer is pulled from the eductor funnel into the eductor valve throat. The partial vacuum is created by the flow of incoming make-up water through the venturi of the eductor throat. Water turbulence within the valve wets out the polymer to enable the formation of quality dispersions.

The eductor outlet should have enough piping (4-5 ft.) to build sufficient back pressure to direct the polymer/ water slurry into an agitated mix tank filled with water to complete polymer hydration.

Eductors can disperse 22.7 kg (50 lb>) of polymer in five to ten minutes with up to 3.0% polymer concentration. Determining the exact eductor and size required for the specific application can best be determined from the technical bulletins and advice of an eductor manufacturer.

Note: Select an eductor which is capable of a solid particle dispersion. Do not use a liquid eductor model. The funnel may be replaced by a flexible hose with stainless steel end tube to enable direct vacuum pick-up from the box of Carbopol® polymer.

Figure 1
Dispersion Rate of Carbopol® Polymers with an Eductor

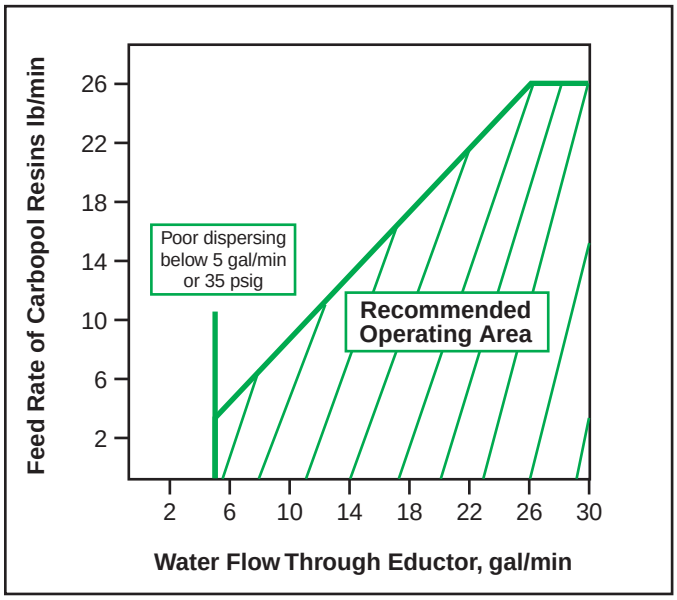
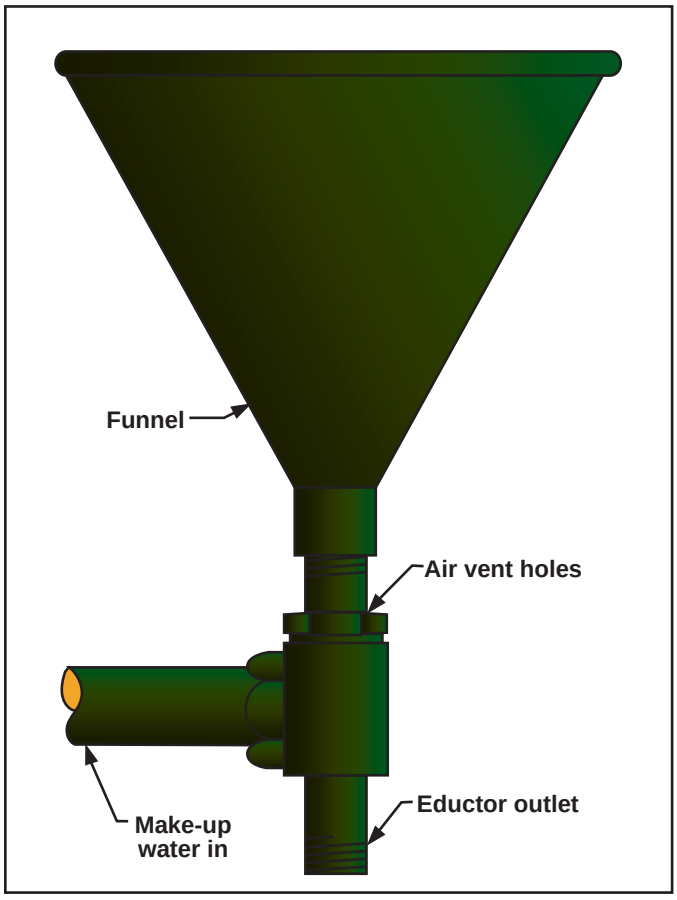


Figure 2
Eductor



Further information regarding eductors can be obtained from these manufacturers:

PENBERTHY
P.O. Box 112
Prophetstown, IL 61277
Phone: (815) 537-2311
FAX: (815) 537-5764

AMETECK INCORPORATED
Division of Schutte and Koerting
2233 State Road
Bensalem, PA 19020
Phone: (215) 639-0900
FAX: (215) 639-1597

FOX VALVE DEVELOPMENT CORPORATION
Hamilton Business Park, Unit 6A
Franklin Road
Dover, NJ 07801
Phone: (201) 328-1011
FAX: (201) 328-3651

DICKENS & WILLIAMSON INCORPORATED
P.O. Box 25009
Charlotte, NC 28212
Phone: (704) 537-0304
FAX: (704) 245-1977

C. Large Quantity Continuous Production Dispersions

The production of good quality, high-solids Carbopol® polymer dispersions from a continuous production scheme is possible with a mechanical in-line powder disperser. Mechanical dispersers use the principle of a high velocity eductor coupled with the mechanical working configuration of an in-line homogenizer to rapidly disperse and wet each individual polymer particle instantaneously. This highly turbulent, high-shear mixer operates at such a rapid rate that the particles are wetted and expelled prior to polymer swelling. Lump formation and polymer degradation from mechanical shearing are eliminated.

The valved powder hopper feed of the mechanical disperser can significantly reduce the level of air entrapment or foaming. Dispersed particles are expelled from the disperser directly in a tank with low/moderate agitation. Table 1 provides comparative outline of mechanical dispersers and their features.

Further information regarding mechanical dispersers can be obtained from the following manufacturers or distributors.

YTRON®
Dr. Karg GmbH
Osternacherstrasse 50
D-8210 Prien am Chiemsee
Germany
Phone: 08051/1457

QUADRO™ PROCESS INC.
72 King Street South
St. Jacobs, ON
Canada NOB 2N0
Phone: (519) 664-3724

SILVERSON MACHINES LTD.
Waterside Chesham, Bucks
HP5 1 PQ, England
United Kingdom
Phone: 0494786331

NORTH AMERICAN
SILVERSON MACHINES LTD.
P.O. Box 589
50 Industrial Drive
East Longmeadow, MA 01028
Phone: (413) 525-4825

ARDE BARINCO INC.
500 Walnut Street
Norwood, NJ 07648
Phone: (201) 784-9880

D. Carbopol® Polymers and Foam

Some foaming can occur in the preparation of Carbopol® polymer in water dispersions, particularly with Carbopol® 1342 and 1382. The significant yield value of these dispersions (even as unneutralized polymers) can result in the formation of a persistent foam.

Table 1
Mechanical Dispersers for Carbopol® Polymers

Model	Manufacturer/ Distributor	Carbopol Polymer Capacity (lbs./hr.)	Maximum Carbopol Polymer Solids (wt. %)	Water Flow Rate (lbs./hr.)
ZC	YTRON QUADRO	1200	15*	15,000
Silverson Flashblend	Silverson Machines Ltd., UK	7500	15-20*	50,000
Arde Vaccuum Dilumelt	Arde Barinco Inc.	1000	4	30,000

* Dispersions of greater than 5 weight percent Carbopol® polymer become dense, solid-like, and impractical to pump or handle.

Tips for Using an Eductor

- Avoid using hot water. The water vapor will prematurely swell the polymer end clog the eductor.
- A minimum 3/4-inch (2 cm) diameter pipe end 35 psig (1.2 atm) water pressure is required for an eductor. Constant pressure must be maintained. If necessary, include a pump for inlet water.
- The eductor should be installed with easily removable plumbing fittings to facilitate cleaning. The discharge must be located above the liquid level of the tank to prevent back pressure.
- Prevent plugging of the eductor throat by starting with a clean eductor and initiating the water flow before adding the preweighed polymer to the eductor funnel. If the eductor does plug with dry polymer, care should be taken when cleaning so that the eductor orifice size is not altered.
- Tapping the funnel manually or with an inexpensive vibrator will prevent Carbopol® polymer from sticking to the funnel.

A handy technique to break the foam is to partially collapse the polymer by the addition of a very low level of strong mineral acid. The yield value is reduced and the trapped air surfaces. Phosphoric or hydrochloric acid is effective at O.5% *on the weight of Carbopol® polymer.* A 1.0 weight percent dispersion of Carbopol® polymer would require 0.005 weight percent or 50 ppm H_3PO_4 or HC1. This level of acid results in no significant contribution of salt when neutralizing Carbopol® polymer. Therefore, the viscosity of the end product is unaffected. Organic acids such as citric or lactic acid are not recommended because they are weaker acids and would yield higher concentrations of residual salts upon neutralization.

II. Indirect Addition of Carbopol® Polymers into Water

In emulsions and alcoholic/water gels, optimum dispersions can best be prepared using the indirect method of adding Carbopol® polymer to a formulation. The dispersion preparation can be simplified by blending Carbopol® polymer and a liquid nonsolvent, such as the oil phase of an emulsion, ethanol/isopropanol, or a hydrophobic surfactant, to form a premix. Any non-solvent of Carbopol® polymer which is part of the product's formulation or is an acceptable additive to the product may be used. A solid concentration of up to 10 weight percent of Carbopol® polymer can work.

A. Oil-in-Water Emulsions

Combine the ingredients of the oil phase and, if required, heat until all materials are liquid. Temperature must not exceed 60°C. Add Carbopol® polymer to the oil phase with moderate to vigorous agitation of 600-800 rpm. Immediately follow with the addition of water containing the appropriate inorganic or amine base to neutralize and thicken the Carbopol® polymer and set the emulsion. Increase the rate of agitation to 1000-1200 rpm and mix for 15-20 minutes or until a smooth, non-grainy texture is apparent.

B. Aqueous Suspension from Insoluble Concentrates of Carbopol® Polymers

Non-solvent dispersions with Carbopol® polymer in mineral oil, mineral spirits, kerosene and low HLB non-ionic surfactants can be prepared using moderate agitation to wet out the polymer prior to its addition into water. This bulk dispersion of the polymer by a non-solvent delays the rate at which the Carbopol® polymer swells or hydrates into water, thus minimizing clumping or the formation of inferior quality aqueous dispersions.

These non-solvent dispersions of Carbopol® polymers should be used immediately to avoid solid polymer settling or agglomeration of the polymer due to plasticization.

C. Dispersing Carbopol® Polymer in Polar Organic Systems

Since polar organic solvents prevent the rapid swelling of Carbopol® polymers, sifting the polymers may be optional. In pure solvent or at high concentrations in water, the Carbopol® polymer can be directly added with minimal agitation. When using pure solvent, add the polymer/solvent slurry to water, or vice versa, with vigorous (800-1200 rpm) mixing. Polar organic solvents include alcohols and glycols.

D. Dry Blending of Carbopol® Polymers

To reduce addition and mixing time, Carbopol® polymers can be initially dry blended with other dry ingredients. The blend is usually free-flowing and free of lumps if the polymer constitutes less than half of the total dry weight. Avoid blending Carbopol® polymer with polyvinylpyrrolidone (PVP), cellulosic polymers or other gums and clays. These blends may give water insoluble complexes or may prevent proper swelling of Carbopol® polymer.

Note: Test the compatibility of Carbopol® polymer in the oil in which it is to be dispersed. In the presence of some polar oils (aromatic or dicarboxylic acid esters) or molten oils above 60°C, Carbopol® polymer will physically plasticize, hindering stable emulsion formation. Dispersing Carbopol® polymer directly into the water in these cases will result in smooth, stable emulsions.