



POLYOX
Water-Soluble Resins
Dissolving Techniques



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POLYOX Water-Soluble Resins Dissolving Techniques

Introduction

Although POLYOX Water-Soluble Resins are totally water-soluble, care must be taken to make sure that they are dissolved completely. Detailed procedures and equipment recommendations for laboratory- and commercial-scale methods for dissolving POLYOX Water-Soluble Resins are described in this book.

The key to dissolution is good initial dispersion.

The most important step in the entire dissolving operation of POLYOX Water-Soluble Resins takes place in the first few seconds—separating the individual resin particles from each other.

High-shear agitators are not necessary and should be avoided.

In water, POLYOX Water-Soluble Resins are instantly wettable. If the POLYOX Water-Soluble Resin powder is not properly dispersed, the partially dissolved, wetted particles will agglomerate and form gels that may never dissolve. High-speed agitation of dissolved POLYOX Water-Soluble Resins must be avoided to prevent shear degradation of the resin. Organic solvents, because of their lower polarity, do not solvate (“wet”) POLYOX Water-Soluble Resins as rapidly as water. As a result, particle agglomeration and gel formation are generally not problems when dissolving POLYOX Water-Soluble Resins in organic solvents.

Three major techniques provide good dispersion of resin particles.

For laboratory or plant requirements, these techniques fall into three major categories:

- Simple direct addition to water.
- Predispersion in water-miscible non-solvents.
- Use of mechanical devices that achieve dispersion with minimal shear.

Which technique you use will depend on the final viscosity and volume required.

For laboratory and less than drum quantities, direct addition of the POLYOX Water-Soluble Resins to water, or non-solvent predispersion of POLYOX Water-Soluble Resins, is recommended. For large volumes or continuous operation, special mechanical devices are available. Each of these techniques is described here.

Direct Addition to Water

The relative ease of dissolving POLYOX Water-Soluble Resins directly in water depends on three factors: (a) rate of viscosity buildup (which is a function of solution concentration and molecular weight), (b) particle size, and (c) type of agitation employed. The underlying factor, once again, is to obtain good resin dispersion before the solution viscosity builds to a point where it is no longer possible to disperse additional resin without high shear. This is best accomplished by adding the POLYOX Water-Soluble Resins at just the “right” rate of addition. If you add it too slowly, the viscosity will build too rapidly, and you will not be able to add the rest of the resin. On the other hand, if you add the resin too rapidly, it will clump up and not dissolve.

Using the Proper Type Stirrer

A one-percent by weight solution of the high molecular weight POLYOX Water-Soluble Resins and a five-percent by weight solution of POLYOX Water-Soluble Resins can be prepared by direct addition of the dry resin to water if the proper type stirrer is used. We have found that either the standard turbine stirrer (see Figure 1) or the standard multi-propeller-type stirrer (see Figure 2) works well to dissolve POLYOX Water-Soluble Resins on a lab scale. These stirrers create a large vortex with only moderate shear. Stir rapidly to create a vortex initially (about 600 rpm), sprinkle in POLYOX Water-Soluble Resins at just the “right” rate, and then decrease the rpm to about 60.

Continue stirring for 30 minutes to one hour until the solution appears homogeneous. In this way, as the viscosity increases, the shear degradation will be minimal. Other concentrations of both POLYOX WSR and WSR N-series may also be prepared by this technique. Depending on the concentration selected and the molecular weight, longer stirring times—up to three hours—may be required.

Hot Water Method

POLYOX Water-Soluble Resins are completely soluble in water at all temperatures, except near the boiling point. At this temperature, POLYOX Water-Soluble Resins precipitate. This phenomenon of inverse solubility provides an alternative technique for dispersing and dissolving POLYOX Water-Soluble Resins. If you choose this method, be careful not to let the steam from the boiling water wet the POLYOX Water-Soluble Resin powder! This may be done, for example, on a lab scale by using a small polypropylene or wooden scoop with curved sides. The curved sides will minimize the contact between the steam and the POLYOX Water-Soluble Resins.

Typically, all the water necessary is heated to near boiling (about 97°C). Condensation will generally appear on the container above the level of the water. If allowed to remain present, undesirable buildup of the POLYOX Water-Soluble Resins may occur. To avoid this, the near-boiling water

should be stirred vigorously to create a vortex (500-600 rpm). This stirring will (1) raise the water level close to the top of the container and reduce or eliminate condensation on the walls and (2) aid in dispersing the POLYOX Water-Soluble Resin particles.

After about one minute, the stirring rate should be reduced to about 50-60 rpm. The source of heat is then removed and a stirring rate of 50-60 rpm is continued until solution is complete. This technique is adaptable to small- and medium-size dissolving operations, where minimal agitation equipment is available.

Figure 1
Standard Turbine Stirrer

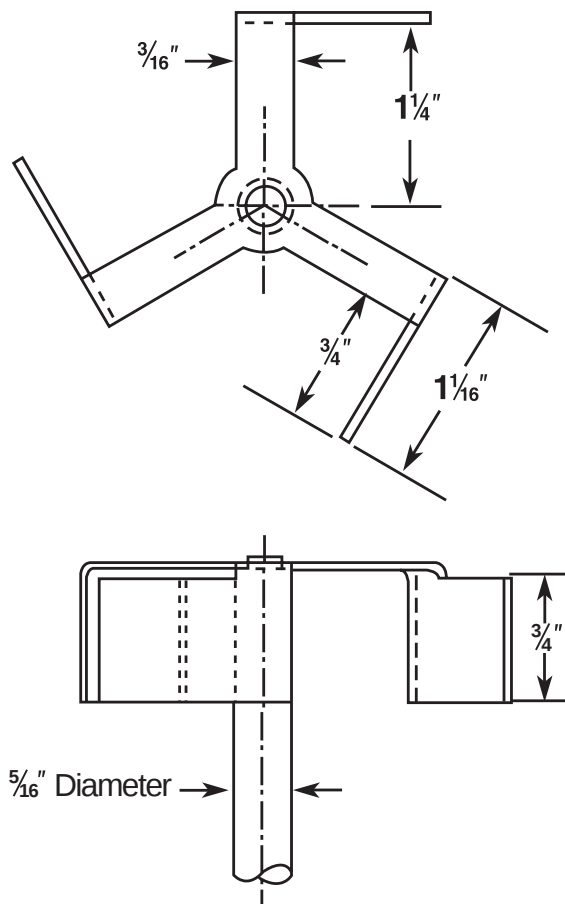
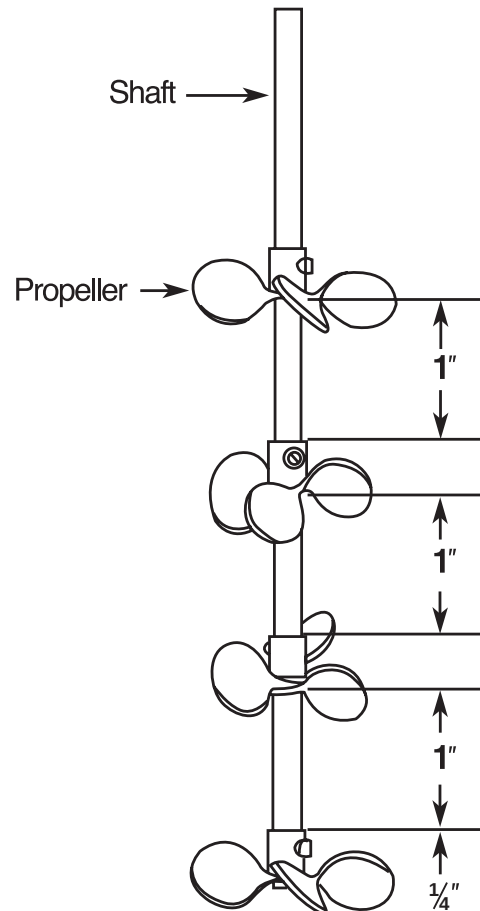


Figure 2
Multi-propeller-type Stirrer



Predispersion in Water-Miscible Non-Solvents

Dispersion of POLYOX Water-Soluble Resins in water-miscible non-solvents separates the individual resin particles from each other. When water is then added, each individual particle has a chance to swell, hydrate, and thereafter dissolve. This technique avoids the typical clumping that occurs if water alone is used. The use of alcohols or glycols also has the added benefit of reducing loss of solution viscosity on aging since these materials behave as solution viscosity stabilizers.

For example, to make a one-percent solution by the predispersion technique, disperse 10 g of POLYOX Water-Soluble Resin in 40 g of anhydrous 2-propanol. Add this slurry to a beaker. Then add 950 g of distilled water all at once. Stir at about 60 rpm and continue stirring for 30 minutes to 3 hours until the solution is homogeneous.

Most non-solvent techniques are suitable for large batch preparation of POLYOX Water-Soluble Resins solutions in plant-scale equipment. For convenience, in plant operation, the non-solvent is added to a stirred vessel. The POLYOX Water-Soluble Resins are then dispersed in the non-solvent followed by addition of the requisite amount of water. Stirring is continued within the guidelines discussed above.

Aqueous Salt and Alkali Solutions

The addition of salts to water will decrease the solubility of the POLYOX Water-Soluble Resins. With such salt or alkali solutions, a similar separation of particles is produced as previously described with organic non-solvents. Dissolution occurs upon dilution with water.

For example, a 1.5-percent solution of POLYOX WSR Coagulant can easily be prepared by first dispersing it in 20-percent sodium hydroxide solution. Specifically, 7.5 g of sodium hydroxide are dissolved in 30 g of distilled water. Then, 15 g of POLYOX WSR Coagulant are dispersed in the concentrated sodium hydroxide solution. Distilled water (955 g) is added to the mixer. With the agitator turning about 600 rpm, the resin slurry is poured in. Agitation is

reduced to about 300 rpm after all the slurry has been added. When the viscosity has become sufficiently high to prevent undissolved resin from settling out, agitation is reduced to about 60 rpm. This rpm is maintained until solution is complete.

Both methods—predispersion in water-miscible non-solvents and dispersion in aqueous salt and alkali solutions—are useful for either large or small operations where agitation facilities are limited, and where the dispersing medium is not objectionable. Table 1 lists examples of other salt solutions useful for dispersing POLYOX Water-Soluble Resins.



Table 1
Salt Solutions Useful for Dispersing POLYOX Water-Soluble Resins

Alkali or Salt	Concentration (Wt Percent)	POLYOX Water-Soluble Resin	Resin in Suspension (Wt Percent)
NaOH	10	WSR Coagulant	15.0
NaOH	20	WSR-301	28.6
		WSR Coagulant	28.6
		WSR-205	37.5
NH ₄ OH	37-39	WSR-301	28.6
		WSR Coagulant	28.6
		WSR-205	37.5
NaCl	Saturated	WSR Coagulant	20.6
		WSR-301	20.6
		WSR-205	20.6
NaCl/NaOH (3/1)	25/20	WSR Coagulant	20.6
NaCl/NaOH (2/1)	20	WSR Coagulant	28.6
NaCl/NaOH (1/1)	20	WSR Coagulant	28.6
MgSO ₄ • xH ₂ O	30	WSR Coagulant	9.1
		WSR-301	9.1
Na ₂ SO ₄	20	WSR Coagulant	9.1
		WSR-301	9.1
		WSR-205	9.1
Na ₃ PO ₄ • 12H ₂ O	20	WSR Coagulant	9.1
		WSR-301	9.1
Na ₂ SO ₃	20	WSR Coagulant	12.2
NaH ₂ PO ₄ • H ₂ O	20	WSR Coagulant	9.1
		WSR-301	9.1
		WSR-205	9.1
Ca(C ₉ H ₃ O ₂) ₂	20	WSR Coagulant	7.4
		WSR-301	7.4
		WSR-205	7.4

Special Mechanical Techniques

The following section provides information on equipment useful for the preparation of drum-size and large-scale quantities of POLYOX Water-Soluble Resin solutions. Dissolving techniques differ, depending on whether POLYOX Water-Soluble Resin powder is used alone or a water-miscible non-solvent is employed.

POLYOX Water-Soluble Resins in Water-Miscible Non-Solvent

Three to 10-percent solutions of POLYOX Water-Soluble Resin can be prepared in small or large quantities by making a 25-percent resin slurry in propylene glycol (or other water-miscible non-solvent). Set up two tandem Moyno pumps: Pump 1 has water only; Pump 2 has the POLYOX Water-Soluble Resin/propylene glycol slurry. Couple both Moyno pumps to a static mixer. The resulting POLYOX Water-Soluble Resin solution will be air bubble-free and have little or no shear degradation.

POLYOX Water-Soluble Resin Powder Alone

Drum-Size Quantities

Equipment: Flocculant Disperser/Eductor

Supplier: Penberthy, Div. of Houdaille Industries
P.O. Box 112
Prophetstown, IL 61277
Tel: 815-537-2311

Capability: 1000-4000 liter batches

Procedure:

1. Weigh 7.5 lb of POLYOX Water-Soluble Resin into a suitable container.
2. Open water valve and add water to 360-gal tank until lower stir blade on the "Lightnin" Mixer is covered.
3. Turn "Lightnin" Mixer on and set at highest speed.
4. Slowly add POLYOX Water-Soluble Resin to the funnel of the Penberthy Flocculant Disperser with water valve full open.
5. Slow the "Lightnin" Mixer to one-half highest speed.
6. Continue adding water until tank is filled to 300-gal mark. If foaming occurs, add water until top of foam is at 300-gal mark. When foam breaks, add additional water to 300-gal mark.
7. Continue mixing at one-half highest speed for 45 min.
8. Dump batch of solution into storage tanks.
9. Stop the "Lightnin" Mixer before the solution level reaches the lower stirrer blade.

Large-Scale Preparation

Equipment:	“Chemix” Mixer - Model DA-4			
Supplier:	Semblex 1635 W. Walnut Street Springfield, MS 65806-1643 Tel: 417-866-1035 Fax: 417-866-0235			
Capability:	150 gal/hr, 0.5% UCARFLOC™ Polymer 309			
Equipment:	Polyelectrolyte Feeding System - Model 35-300			
Supplier:	Wallace and Tiernan Division of Pennwalt Corporation 642 Broad Street Clifton, NJ 07013 Tel: 201-472-7300			
Capability:	1.8 gal/hr			
Supplier:	Wallace and Tiernan GmbH Postfach 1563 D-8870 Gunzburg, Germany Tel: 011-49-8221-9040 TX: 531155			
Equipment:	“Polymair” Model 500-200 - Fully Automatic Packaged Processing System			
Supplier:	Acrison, Inc. 20 Empire Blvd. Moonachie, NJ 07074 Tel: 201-440-8300 TX:134395		984 Route #9 Parlin, NJ 08859 Tel: 201-721-3240 TX:844490	
Capability:	15 lb polymer/hr of 0.3% solution. Utilizing a 20-min cycle, approximately 10 gal/min of solution. 200 gal of 0.3% UCARFLOC Polymer 309 every 20 min, 24 hr/day, 7 days/wk			
Equipment:	“Polyblend” Models DP-200, DP-500, DP-1500			
Supplier:	Stranco, Inc Route 50 North P.O. Box 389 Bradley, IL 60915 Tel: 1-800-882-6466 Fax: 815-939-9845		Stranco, Ltd. Commercial Road Eastbourne, E. Sussex BN 21 3XE, England Tel: 011-44-323-646163 Fax: 011-44-323-646035	
Capability:	Model	Lb/hr	U.S. Gal/hr of 0.25% Solution	U.S. Gal/hr of 0.1% Solution
	DP-200	12.5	600	1500
	DP-500	25	1200	3000

Grades of POLYOX Water-Soluble Resins

POLYOX Water-Soluble Resins are supplied in a wide variety of molecular weight grades and formulated compounds in standard and NF (National Formulary) grades. Table 2 shows the current product range and their corresponding molecular weights and properties. These resins are classified in two series: WSR and WSR N.

Table 2: Grades of POLYOX Water-Soluble Resins

POLYOX Grade	Approximate Molecular Weight ⁽¹⁾
WSR N-10	100,000
WSR N-80	200,000
WSR N-750	300,000
WSR N-3000	400,000
WSR-205	600,000
WSR-1105	900,000
WSR N-12K	1,000,000
WSR N-60K	2,000,000
WSR-301	4,000,000
WSR Coagulant	5,000,000
WSR-303	7,000,000
WSR-308	8,000,000
UCARFLOC Polymer 300	4,000,000
UCARFLOC Polymer 302	5,000,000
UCARFLOC Polymer 304	7,000,000
UCARFLOC Polymer 309	8,000,000
POLYOX Water-Soluble Resins NF for Pharmaceutical Applications⁽³⁾	
WSR N-10 NF	100,000
WSR N-80 NF	200,000
WSR N-750 NF	300,000
WSR-205 NF	600,000
WSR-1105 NF	900,000
WSR N-12K NF	1,000,000
WSR N-60K NF	2,000,000
WSR-301 NF	4,000,000
WSR Coagulant NF	5,000,000
WSR-303 NF	7,000,000

The physical property data listed are considered to be typical properties, not specifications.

(1) Based on rheological measurements. Molecular weights obtained by other methods, including light scattering and gel permeation chromatography, may not be directly comparable.

(2) Model RVT

(3) POLYOX Water-Soluble Resins-NF comply with The National Formulary standard for "Polyethylene Oxide" (page 2285-6) in USP 23-NF 18 issued in 1995.

Viscosity Range in mPa • sec (cP) for Various Weight Percentages of Aqueous Solutions at 25°C			Brookfield Viscometer, Model RVF, Spindle No./Speed, rpm
5%	2%	1%	
12–50	—	—	1/50 ⁽²⁾
65–115	—	—	1/50 ⁽²⁾
600–1000	—	—	1/10
1000–1200			2/10
2250–4500	—	—	1/2
4500–8800	—	—	2/2
8800–17,600	—	—	2/2
—	400–800	—	1/10
—	2000–4000	—	3/10
—	—	1650–5500	2/2
—	—	5500–7500	2/2
—	—	7500–10,000	2/2
—	—	10,00–15,000	2/2
—	—	1650–5500	2/2
—	—	5500–7500	2/2
—	—	7500–10,000	2/2
—	—	10,000–15,000	2/2
30–50			1/50 ⁽²⁾
55–90			1/50 ⁽²⁾
600–1200			1/10
4500–8800			2/2
8800–17,600			2/2
	400–800		1/10
	2000–4000		3/10
		1650–5500	2/2
		5500–7500	2/2
		7500–10,000	2/2

Typical Physical Properties

POLYOX Water-Soluble Resins are produced and supplied as white, granular powders, freely soluble in water and possessing a slightly ammoniacal odor. Typical properties are given in Table 3.

Table 3
Typical Physical Properties⁽¹⁾ of POLYOX Water-Soluble Resins

Appearance	Off-white powder
Crystalline Melting Point ⁽²⁾ (X-ray and DSC), °C	62–67
Odor	Slightly ammoniacal
Melt Flow Temperature, °C	> 98
Volatiles Content, as packaged, % by wt (at 105°C)	< 1.0
Alkaline Earth Metals, % by wt as CaO, max	1.0
Powder Bulk Density, lb/ft ³ (kg/m ³)	19–37 (304–593)
Polymer Density, g/cc	1.15–1.26
Moisture Content, as Packaged, %	< 1
Heat of Fusion, cal/gm	33
Solution pH	8–10
Particle Size, % by wt	
Average through 10-mesh (U.S. Standard)	100
Average through 20-mesh	96

(1) Data listed here are considered to be typical properties, not specifications.

(2) At temperatures far above the crystalline melting point, high polymers of POLYOX Water-Soluble Resins still retain a very high degree of crystalline character.



Product Safety

When considering the use of any Dow products in a particular application, you should review our latest Material Safety Data Sheets and ensure that the use you intend can be accomplished safely. For Material Safety Data Sheets and other product safety information, contact us at the numbers listed on the back cover of this brochure. Before handling any other products mentioned in the text, you should obtain available product safety information and take necessary steps to ensure safety of use.

**For more information, complete literature, and product samples,
you can reach a Dow representative at the following numbers:**

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