SEKISUI

Solution Preparation Guidelines

SELVOL™
POLYVINYL ALCOHOL

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About the Company

The Sekisui Chemical Group is a global company that operates in three major businesses: High Performance Plastics, Urban Infrastructure and Environmental Products, and Housing. Founded in 1947 and currently headquartered in Osaka and Tokyo, Japan, Sekisui strives to deliver a wide range of products and services to enrich people's lives and the social infrastructure.


Architectural Glass


Urban Infrastructure and Environmental Product

Our Promise

Through prominence in technology and quality, Sekisui Chemical Group will contribute to improving the lives of the people of the world and the Earth's environment, by continuing to open up new frontiers in residential and social infrastructure creation and chemical solutions.


Housing

Sekisui Specialty Chemicals

Sekisui produces and sells one of the most complete lines of polyvinyl alcohol in the world. Since the introduction of Selvol Polyvinyl Alcohol, we have developed a high level of expertise in both the production and use of PVOH.

Based in Dallas, Texas, Sekisui Specialty Chemicals is a leading polyvinyl alcohol supplier with manufacturing facilities in Calvert City, Kentucky, Pasadena, Texas and Tarragona, Spain. The combined capacity of the three plants makes Sekisui a leading global merchant supplier of polyvinyl alcohol.

Sekisui's commitment to polyvinyl alcohol is especially evident in our research and applications support activities. We have one of the largest technical services, product application, and analytical services groups in the world. Research and application development is carried out at our facilities in Houston, Texas. Sekisui also has a global sales force located in offices worldwide to respond more quickly to the needs of our customers.

SELVOL™ POLYVINYL ALCOHOL

Introduction

This brochure is intended to serve as guidelines to proper solution of Selvol Polyvinyl Alcohols.
For more detailed information on specific applications, the preparation of polyvinyl alcohol solutions, please refer to our other brochures, visit our website at www.selvol.com, or call our Product Information Center at +1-281-280-3460.

Environmental, Health, and Safety

Please refer to our Material Safety Data Sheets (MSDSs) or Safety Data Sheets (SDSs) for information on the safe use and handling of Selvol Polyvinyl Alcohol, including toxicity, fire, and explosion hazards, as well as environmental effects. An MSDS can be obtained online at www.selvol.com.

FDA Compliance

Polyvinyl alcohol is used in many food contact applications, including food packaging adhesives and coatings for paper and paperboard. For more specific information on the FDA status of any of our grades, please contact our Product Information Center at +1-281-280-3460.

TABLE 1:
Selvol Polyvinyl Alcohol Right-to-Know Information

Ingredient	CAS Number
Selvol Polyvinyl Alcohol • Super and Fully Hydrolyzed • Partially and Intermediate Hydrolyzed	 9002-89-5 25213-24-5
Water	7732-18-5
Methanol	67-56-1
Sodium Acetate	127-09-3

Selvol Polyvinyl Alcohol

Selvol Polyvinyl Alcohol is a white, granular, water-soluble resin manufactured by polymerizing vinyl acetate and hydrolyzing the resultant polymer to produce the alcohol (Figure 1).

Because PVOH is synthesized from polyvinyl acetate, a variety of different grades of Selvol Polyvinyl Alcohol is available that varies in molecular weight and hydrolysis level. These two factors are the major determinants of the performance properties of PVOH.

FIGURE 1:
General Structure of Polyvinyl Alcohol

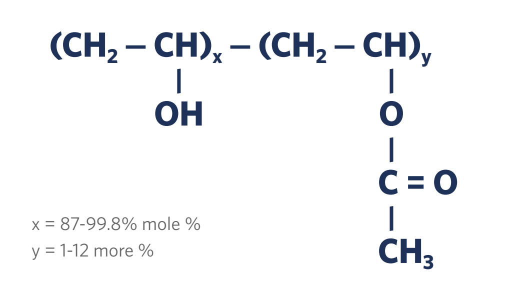


TABLE 2:
Selvol Polyvinyl Alcohol Molecular Weight

Viscosity[1] (cP)	Viscosity Type	Degree of Polymerization	Average Weight Molecular Weight Range	Number Average Molecular Weight Range
3-6	Low	150 - 650	13,000 - 50,000	7,000 - 23,000
22-30	Medium	1000 - 1500	85,000 - 124,000	44,000 - 65,000
45-72	High	1600 - 2200	146,000 - 186,000	70,000 - 101,000

1 4% aqueous solution viscosity.

Molecular weight is a measure of polymer chain length and is typically reported as a 4% aqueous solution viscosity (Table 2).

Molecular Structure PVOH

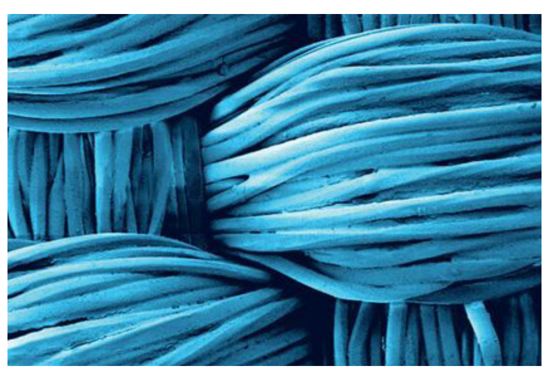
Table 3:
Description of the Different Hydrolysis Levels for PVOH

Grade	Hydrolysis Mole %
Super	99.3+
Fully	98.0-98.8
Intermediate	90.0-97.0
Partially	87.0-89.0

Hydrolysis level is a measure of the mole % hydroxyl functionality on the polymer. The hydrolysis level of PVOH is typically categorized as shown in Table 3.

Polyvinyl Alcohol Business

Important end-use markets for these polymer products include textiles, paper, adhesives, building products, and specialty applications. Selvol Polyvinyl Alcohol resins perform well as textile sizing agents, pigment binders, emulsifying agents, and in adhesive and protective film applications. Special properties may be imparted by blending grades or compounding with other ingredients.



Selvol Polyvinyl Alcohols are widely used for textile warp sizing. Selvol Polyvinyl Alcohol films exhibit high abrasion resistance, elongation, tensile strength, and flexibility. Partially hydrolyzed grades, which possess increased polyester adhesion, can lead to superior abrasion resistance and weavability.

Additionally, partially hydrolyzed polyvinyl alcohols will desize far easier than other grades providing numerous benefits in textile finishing operations. Selvol Polyvinyl Alcohols are also used by the paper industry for surface sizing and coating. Selvol PVOH significantly improves grease, solvent and water resistance as well as web strength. Selvol Polyvinyl Alcohol products are also well suited as binders in pigmented coatings systems and as carriers for optical brighteners.

In adhesive formulations Selvol Polyvinyl Alcohols can function as the primary binder or a compounding agent. They bond particularly well to cellulosic surfaces and offer improved water resistance, strength, and resistance to grease and other petroleum hydrocarbons.

Polymerizers also find Selvol Polyvinyl Alcohols valuable as a dispersing/stabilizing agent. They behave primarily as a protective colloid and often enhance the emulsifying action at very low concentrations.

Selvol Polyvinyl Alcohols are used in a wide variety of other industrial applications such as a temporary binder for ceramics, water soluble films, strippable coatings, and nonwovens. The products also find use in various building products such as joint cements and mortars.

Solubility

All Selvol Polyvinyl Alcohol grades are readily soluble in water. Other solvents include dimethyl sulfoxide, acetamide, glycols, and dimethylformamide. Conditions for the dissolution are governed primarily by degree of hydrolysis, but they are influenced by other factors such as molecular weight, particle size distribution, and particle crystallinity.

Optimum solubility occurs at 87-89% hydrolysis. Grades in this range exhibit a high degree of cold water solubility. For complete dissolution, however, these grades require temperatures of 185°F (85°C), with a hold time of 30 minutes.

Higher hydrolysis grades, including the intermediate, fully, and super grades, require progressively more energy to dissolve because of their greater degree of crystallinity. For dissolution, these grades require temperatures in the range of 200-205°F (93-95°C), with a hold time of 30 minutes (see Figure 2).

Once in solution, only partially hydrolyzed grades are viscosity stable with time. Super and fully hydrolyzed Selvol Polyvinyl Alcohol grades will tend to thicken somewhat through hydrogen bonding. Viscosity relationships are portrayed as a function of concentration at various temperatures in Figure 3. These viscosities are for general guidelines only. Actual values will vary due to differences in the testing method.

FIGURE 2:

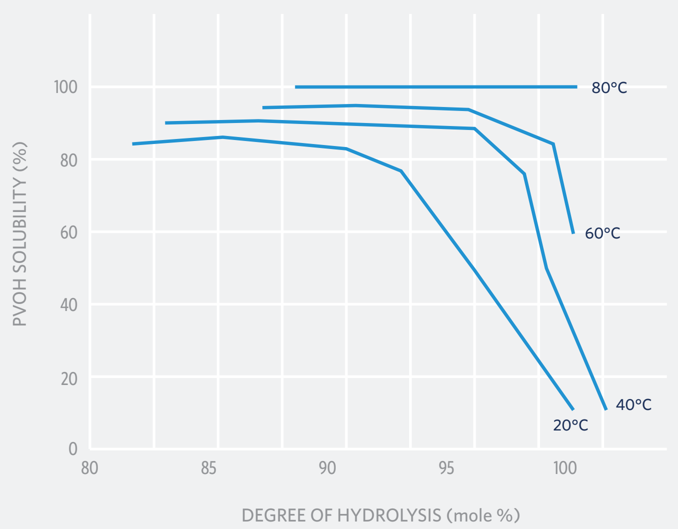
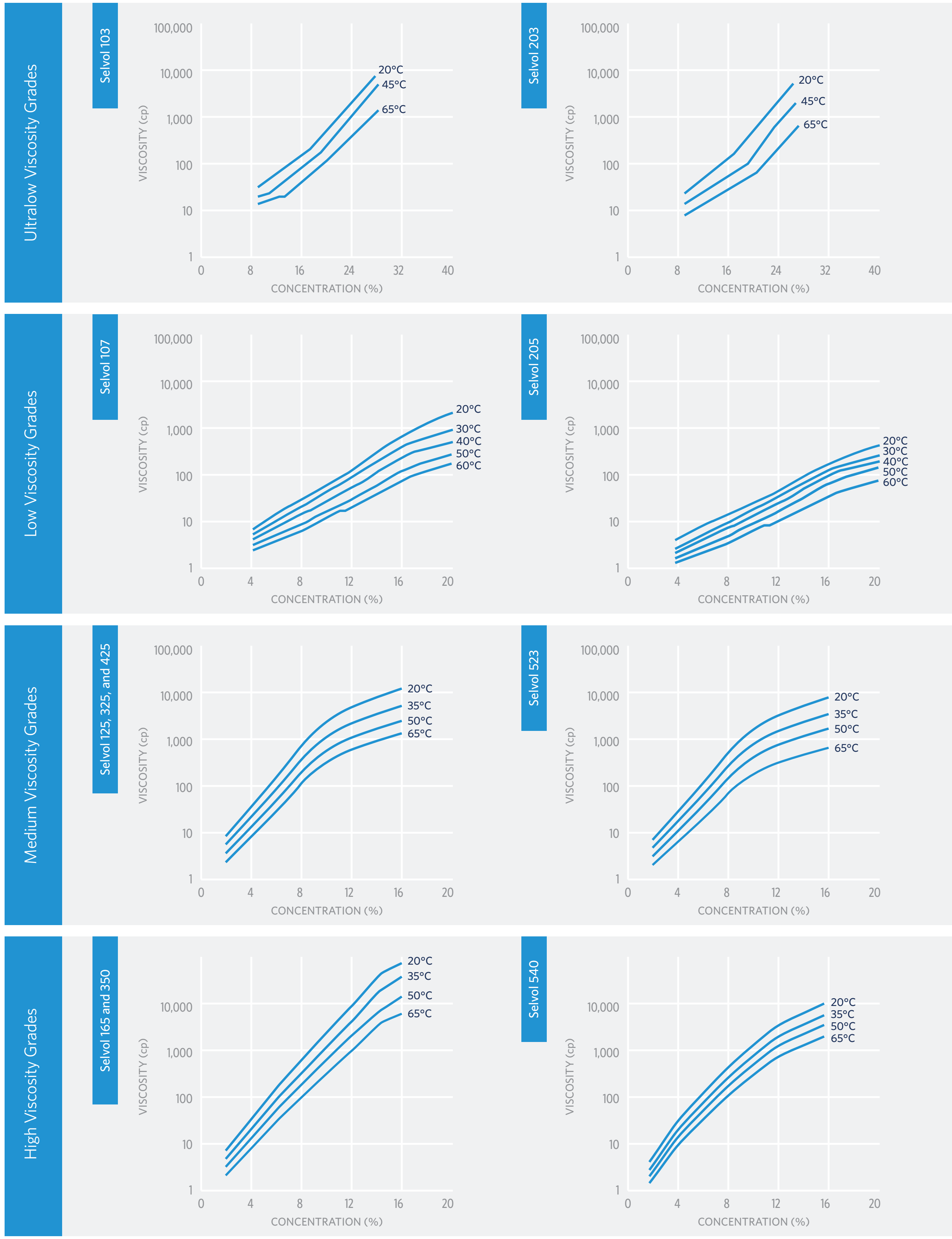


FIGURE 3:
How the Concentration of Selvol Polyvinyl Alcohol Affects Solution Viscosity*



General Guidelines

The most critical step in effectively dissolving polyvinyl alcohol is to completely disperse the particle in water. Since the surface of the particles will swell very quickly and then clump together, it is very important to add the granule slowly, using good agitation, to cool water (<100°F/38°C).

Note that good agitation does not mean high shear. It means sufficient agitation to disperse the particles, without whipping air into the solution.

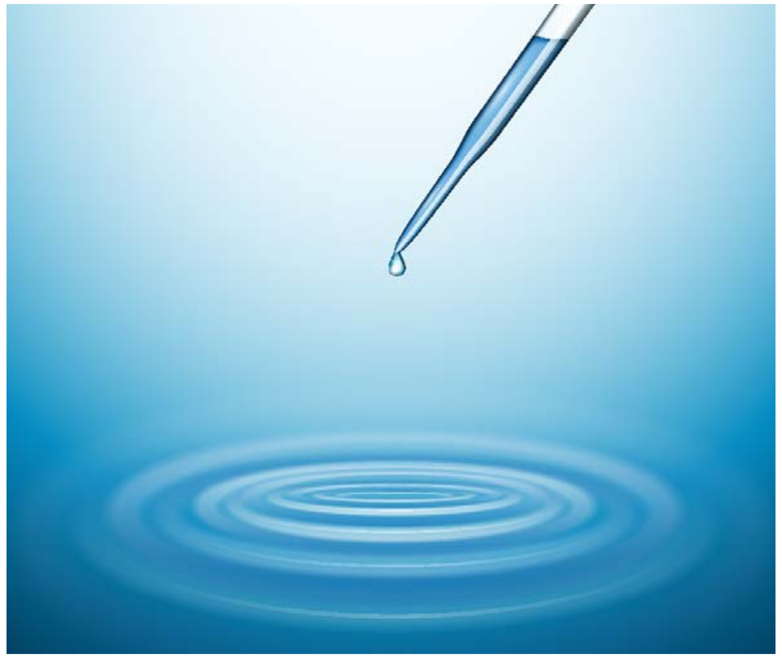


TABLE 5:
Recommended Defoamers for Use with PVOH Solution*

Defoamer	Supplier	Use Level
FC 407	ESP Enterprises	<1% d/d
Antifoam 116	Harcros	< 1% d/d
Industrol DF 132	BASF	< 1% d/d

* Note: The FDA compliance status of the recommended additives should be verified with the respective manufacturer.

Cool water is important, particularly for the partially hydrolyzed grades, to allow good dispersion before particle swelling. If polyvinyl alcohol is added to hot water, the particles swell rapidly and clump together before complete dissolution can be achieved. Once the granules are well dispersed in cool water, the suspension can be heated. Partially hydrolyzed grades should be heated to at least 185°F (85°C); fully and super hydrolyzed grades should be heated to at least 205°F (95°C). The cook solution should be held at these temperatures for at least half an hour. See pages 7 and 8 for step-by-step solution preparation instructions.

UNDISSOLVED PARTICLES

Undissolved polyvinyl alcohol particles will appear as transparent gels when incompletely cooked. They are not easily visible just by peering into the cook vessel, nor are they easily filtered out. If polyvinyl alcohol is not fully solubilized, it will not achieve its optimum performance. Complete solubility, however, is easy to achieve if you follow a consistent cooking procedure based on the recommendations in this brochure.

FOAM

The tendency for polyvinyl alcohol to generate foam is highly dependent upon the degree of hydrolysis, and to a lesser extent, on the mechanical dynamics unique to each preparation and end-use process. Generally, the higher the hydrolysis, the less tendency to foam. Fully and super hydrolyzed grades may be used without defoamers, whereas intermediate and partially hydrolyzed grades nearly always require a defoamer. See Table 5 for recommended defoamers.

BIOCIDES

If polyvinyl alcohol solutions are held for more than 24 hours, a biocide addition is recommended. See Table 6 for recommended biocides.

TABLE 6:
Recommended Biocides for Use with PVOH Solutions*

Biocide	Supplier	Use Level
Kathon LX	Dow Chemical	< 50 ppm
Dowicil 75	Dow Chemical	1000 - 2000 ppm

* Note: The FDA compliance status of the recommended additives should be verified with the respective manufacturer.

FIGURE 4:
Determination of Polyvinyl Alcohol Addition for Desired Solution Solids Content

$$\text{Polyvinyl Alcohol Addition (dry wt.)} = \frac{X * Y}{100\% - \%\ \text{Total Volatiles}^{1}}$$

X = Desired solution solids content[2]   Y = Net weight of final solution

EXAMPLE:
To make 100 grams of a 20% solution of Selvol 205, add the following amount of polyvinyl alcohol.
The % Total Volatiles for your current lot of Selvol 205 is 4.5%

X = 20%
Y = Net weight of final solution

$$\text{Polyvinyl Alcohol Addition (dry wt.)} = \frac{(20/100) * 100\ \text{grams}}{(100\% - 4.5\% / 100)} = 20.94\ \text{grams}$$

[1] The specification for % Total Volatiles is 5.0% maximum. Refer to the C of A for actual value.
[2] For best results do not exceed maximum solids guidelines (see Table 8)

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Solution Preparation by Live Steam, Jacketed Vessels or Immersed Coils

1. Fill tank with ***unheated*** water. All lines should be free of borax and other containments.
2. Turn on mixer. Surface of water should move vigorously. Top blade should be submerged to half the height of the water.
3. If using a defoamer, add it at this point, prior to any polyvinyl alcohol addition.
4. Add polyvinyl alcohol to unheated water with agitation. Recommended rates of addition depend on the grade of polyvinyl alcohol. See Table 8.
5. For best results, do not exceed maximum solids guidelines. See Table 8.
6. Elevate solution temperature to 185- 205°F (85-95°C). See Table 8. If using live steam, allow 15-20% condensation from the steam injection.
7. Upon reaching cook temperature, hold at temperature for 30 minutes.
8. Selvol Polyvinyl Alcohol is now in solution. It can be used at any temperature.
9. If the Selvol Polyvinyl Alcohol Solution is stored for more than 24 hours, a biocide addition is recommended.

For ready Selvol Polyvinyl Alcohol Solutions available for purchase please refer to Table 7.

TABLE 7:
Ready Selvol Polyvinyl Alcohol Solutions Available for Purchase

STANDARD GRADES							
Grade	Hydrolysis, % min.	Solution Viscosity, cP	Solution Solids, wt%	Solution pH	Total Volatiles, wt% Max	Ash - ISE, % Max[5]	Methanol, wt% Max
Super Hydrolyzed							
Selvol PVOH 08-125	99.3+	100-1300	7.0-8.0	5.5-7.5	1.0	1.2	0.9
Selvol PVOH 15-103	98.0-98.8	50-500	14.5-15.5	5.0-7.0	1.0	1.2	0.9
Selvol PVOH 09-325	98.0-98.8	200-1300	8.0-9.0	5.5-7.5	1.0	1.2	0.9
Selvol PVOH 24-203	87.0-89.0	250-1300	23.0-25.0	4.5-6.5	1.0	0.9	0.9
Selvol PVOH 09-523	87.0-89.0	250-1300	8.50-9.50	4.5-6.5	1.0	0.9	0.9
Selvol PVOH 21-205	87.0-89.0	800-1300	20.0-22.0	4.5-6.5	1.0	0.7	0.9

Note:
- Ash, hydrolysis, methanol, and total organic volatiles are based on the polyvinyl alcohol from which the solution was prepared.
- pH solids and viscosity are based on the solution.

Solution Preparation by Jet Cooker

1. Fill slurry tank with ***unheated*** water. All lines should be free of borax and other containments.
2. Turn on mixer. Surface of water should move vigorously. Top blade should be submerged to half the height of the water.
3. If using a defoamer, add it at this point, prior to any polyvinyl alcohol addition.
4. Add polyvinyl alcohol to unheated water with agitation. Recommended rates of addition depend on the grade of polyvinyl alcohol. See Table 8. To compensate for steam dilution, the initial concentration of Selvol Polyvinyl Alcohol should be higher than the required concentration.
5. For best results, do not exceed maximum solids guidelines. See Table 8.
6. Make sure all filters between the tank and jet, and the jet orifice and retention coil have a minimum hole diameter of 0.0185 in (4.75mm, U.S. Series Equivalent 4).
7. Pump slurry through the jet.
8. Jet temperature of 200-350°F (93-177°C) should be used. Steam pressure should be maintained above 25 psig.
9. Residence coil of 5-10 minutes is strongly recommended.
10. Allow for a 2% solids drop coming out of jet.
11. Unlike starch, polyvinyl alcohol does not instantaneously solubilize and must be kept in the hold vessel for 15 minutes prior to use. Solution temperatures in hold vessel should be maintained at 205°F for super hydrolyzed grades, 200°F for fully and intermediate hydrolyzed grades, and 185°F for partially hydrolyzed grades.
12. Water dilutions should take place after the 15 minute hold time.
13. If the Selvol Polyvinyl Alcohol Solution is stored for more than 24 hours, a biocide addition is recommended.

For ready Selvol Polyvinyl Alcohol Solutions available for purchase please refer to Table 7.

To determine desired solution solids content for Selvol Polyvinyl Alcohol please refer to calculations in Figure 4.

TABLE 8:
Solution Preparation

Grade	Addition Rate	Maximum Recommended Solids	Minimum Cook-Out Temperature
Super Hydrolyzed			
Selvol PVOH 125	10 sec/bag	10%	205°F (96°C)
Selvol PVOH 165	10 sec/bag	7%	205°F (96°C)
Fully Hydrolyzed			
Selvol PVOH 103/E 103	10 sec/bag	25%	200°F (93°C)
Selvol PVOH 107/E 107	10 sec/bag	20%	200°F (93°C)
Selvol PVOH 310/E 310	10 sec/bag	15%	200°F (93°C)
Selvol PVOH 325/E 325	10 sec/bag	10%	200°F (93°C)
Selvol PVOH 325LA	10 sec/bag	10%	200°F (93°C)
Selvol PVOH 350	10 sec/bag	7%	200°F (93°C)
Intermediate Hydrolyzed			
Selvol PVOH 418	1 min/bag	12%	195°F (91°C)
Selvol PVOH 425	1 min/bag	10%	195°F (91°C)
Selvol PVOH 443	1 min/bag	7%	195°F (91°C)
Partially Hydrolyzed			
Selvol PVOH 502	2 min/bag	30%	185°F (85°C)
Selvol PVOH 203/E 203	2 min/bag	30%	185°F (85°C)
Selvol PVOH 504	2 min/bag	30%	185°F (85°C)
Selvol PVOH E 4/88LA*	2 min/bag	30%	185°F (85°C)
Selvol PVOH E 5/88LA*	2 min/bag	20%	185°F (85°C)
Selvol PVOH 205/E 205	2 min/bag	20%	185°F (85°C)
Selvol PVOH E 8/88	2 min/bag	20%	185°F (85°C)
Selvol PVOH 508	2 min/bag	20%	185°F (85°C)
Selvol PVOH 513	2 min/bag	15%	185°F (85°C)
Selvol PVOH 518	2 min/bag	15%	185°F (85°C)
Selvol PVOH 523/E 523	2 min/bag	10%	185°F (85°C)
Selvol PVOH 540	2 min/bag	7%	185°F (85°C)
Warp Size			
Selvol PVOH WS-724	1 min/bag	15%	195°F (91°C)
Selvol PVOH WS-53NF	1 min/bag	10%	185°F (85°C)
Polymerization			
Selvol PVOH 805	2 min/ bag	20%	185°F (85°C)
Selvol PVOH 818	2 min/bag	15%	185°F (85°C)
Selvol PVOH 823	2 min/ bag	10%	185°F (85°C)
Selvol PVOH 830	2 min/bag	10%	185°F (85°C)
Selvol PVOH 840	2 min/ bag	7%	185°F (85°C)
Fine Particle (S-Grade)			
Selvol PVOH 165SF	10 sec/bag	7%	Room Temperature
Selvol PVOH 203S9/E 203S	2 min/bag	30%	Room Temperature
Selvol PVOH 205S/E 205S	2 min/bag	20%	Room Temperature
Selvol PVOH 513S	2 min/bag	15%	Room Temperature
Selvol PVOH 523S/E 523S	2 min/bag	10%	Room Temperature
Selvol PVOH 540S	2 min/bag	7%	Room Temperature
Tackified Grades			
Selvol PVOH MH-82		10%	
Selvol PVOH MM-51		10%	
Selvol PVOH MM-81		10%	

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Equipment

VESSEL

A vessel for dissolving Selvol Polyvinyl Alcohol should be constructed of 304 stainless steel or other corrosion-resistant materials to ensure contamination-free solutions. A height-to-diameter ratio of 1.0 is common.

Baffles, which help to reduce particle descent and prevent severe vortex formation, should be positioned 90 degrees apart and extend the entire height of the vessel.

It is recommended that the baffles width be 1/12 x the vessel diameter and that the offset from the wall be 1/72 x the diameter. The bottom of the vessel must be equipped with a flush valve to prevent particles from settling into and plugging the discharge line. Alternatively, a recycle loop may be employed off the bottom discharge line below the surface to minimize foam. See Figure 6 for recommended vessel design.

AGITATION

Adequate agitation is required for both dispersing particles and for proper heat transfer. The agitation system generally consists of either one or two pitched turbine impellers. These impellers are most effective when they extend to 50-70% of the vessel diameter and are adjusted to a speed of 60-80 rpm. A variable speed agitator is useful in finding the optimum rpm level, and it can better accommodate viscosity changes during polymer dissolution. See Figure 6 for recommended guidelines on agitation.

HEAT

Heat can be transferred to the solution via coils, jacket or the most widely preferred, direct steam injection (live steam injection or steam jet cooker).

LIVE STEAM INJECTION

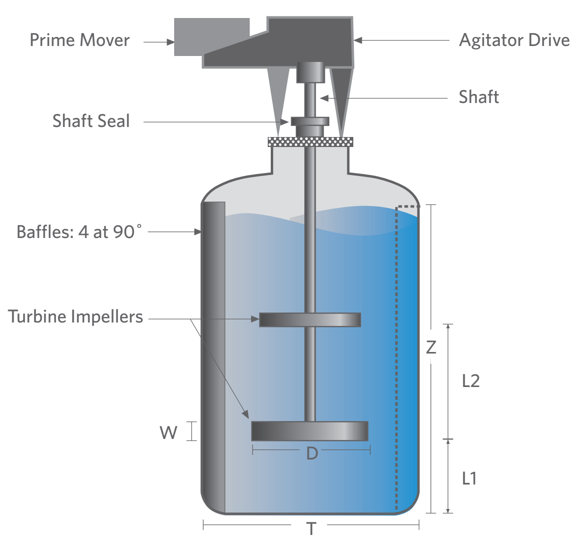
Direct steam injection allows shorter heating times than indirectly heated jacketed or immersion coiled vessels. However, solution concentration is more difficult to control. Since 15-20% of the water content of the batch may come from condensed steam, care must be taken to allow for the dilution effect on the final concentration of the solution. Boiler treatment chemicals or other impurities that might be present in steam could have an adverse effect on the solution.

Steam coils should enter through the side or bottom of the vessel, and steam pressure should be 15-25 psig to avoid localized water evaporation and baking of the resin at the steam inlet.

JACKETED VESSELS

Jacketed vessels allow good control of the solution concentration, since no steam condensate dilutes the final solution. The heating jacket should cover only the bottom half to bottom third of the vessel. To prevent heat transfer loss, reduce concentration and cleaning problems, the temperature differential from the jacket to the vessel should be kept low to avoid baking the Selvol Polyvinyl Alcohol onto the hot vessel wall. Low pressure steam at 15-25 psig is recommended. Good agitation reduces the possibility of fouling.

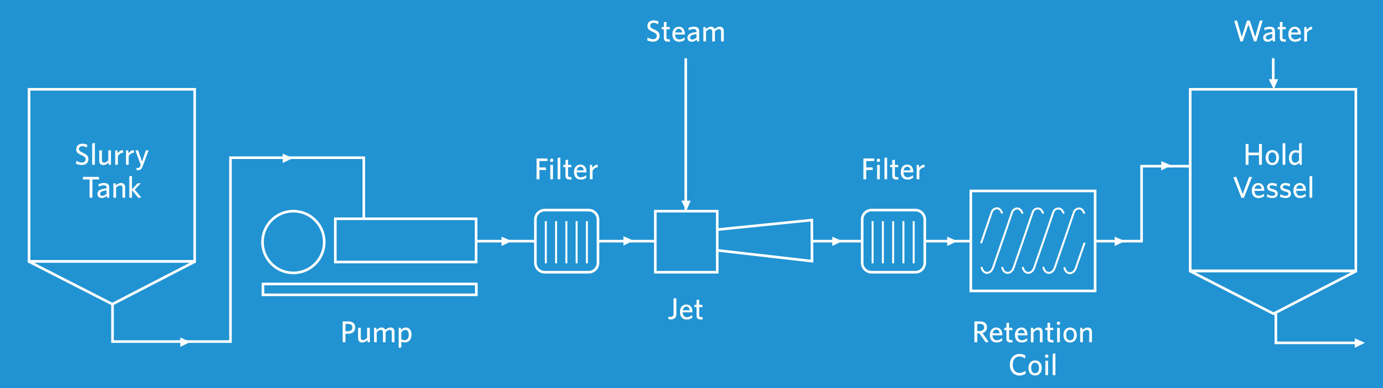
FIGURE 5:
General Design Considerations for Dissolving Systems



		SINGLE IMPELLER	DOUBLE IMPELLER
N	Agitator Speed (rpm)	$1300/(V)^{0.4}$	$1640/(V)^{0.4}$
T	Tank Diameter (ft)	$\sqrt[3]{0.17\ V}$	$\sqrt[3]{0.085\ V}$
D	Impeller Diameter (ft)	T/2	T/2
W	Impeller Width (ft)	D/8	D/8
P	Agitator Power (horsepower)	$2.45 \times 10^{-8}\ N^3\ D^5$	$5.0 \times 10^{-8}\ N^3\ D^5$
L1	Height of Bottom Impeller from Tank Bottom (ft)	Z/4	T/4
L2	Height of Top Impeller from Tank Bottom (ft)		$(2/3)\ Z$
V	Vessel Volume (gallons)		
Z	Liquid Height in Tank (ft)		

Process Vessel and Major Components of a Double Impeller Turbine Agitator

FIGURE 6:
Typical Design for Steam Jet Cooking Systems



IMMERSION COILS

Immersion coils allow good control of the solution concentration. If immersion coils are used, the temperature of the coils should be kept below 240°F. Excessive heat may cause localized water evaporation, leading to polymer film formation on the heated surface. This film may flake off and cause subsequent problems. Immersion coils, if fouled, are not as easily cleaned as are jacketed vessels.

STEAM JET COOKER

High pressure steam jet cookers are designed and manufactured by starch suppliers for use in dissolving starch. While most Selvol Polyvinyl Alcohol customer's batch cooks, a small number of customers prefer to jet cook. When jet cooking Selvol Polyvinyl Alcohol, it is important to remember two critical differences between polyvinyl alcohol and starch:

1. Selvol Polyvinyl Alcohol has a much larger particle size than starch.
2. Unlike starch, Selvol Polyvinyl Alcohol does not instantaneously dissolve when impinged with high pressure/temperature steam. Therefore, additional heat and time are required to solubilize polyvinyl alcohol after the jet.

Note: Prior to purchasing your cooking system, please contact your Sekisui representative. Many of the design considerations will be based on the grade being dissolved and on your specific requirements.

Typically, Selvol Polyvinyl Alcohol is slurred in an agitated vessel. To cook the solution, the slurry is pumped from the slurry tank through a mixing head into which steam is injected (see −figure 6). Steam pressure should be maintained above 25 psig. As pressure builds up in the mixing head, it acts like a pressure cooker, resulting in a substantial reduction in time required for dissolution as compared with conventional methods.

To ensure complete dissolution of all polyvinyl alcohol particles, a residence coil (5 to 10 minutes) and hold vessel are strongly recommended.

The holding vessel should be insulated and equipped with a heat source to maintain solution temperatures of 205°F for super hydrolyzed grades, 200°F for fully and intermediate hydrolyzed grades, and 185°F for partially hydrolyzed grades.

All in-line −filter screens, before and after the jet, must be adequately sized to allow all the swollen resin particle to pass through. The filters should have a minimum hole diameter of 0.0185 in (4.75mm, U.S. Series Equivalent 4).

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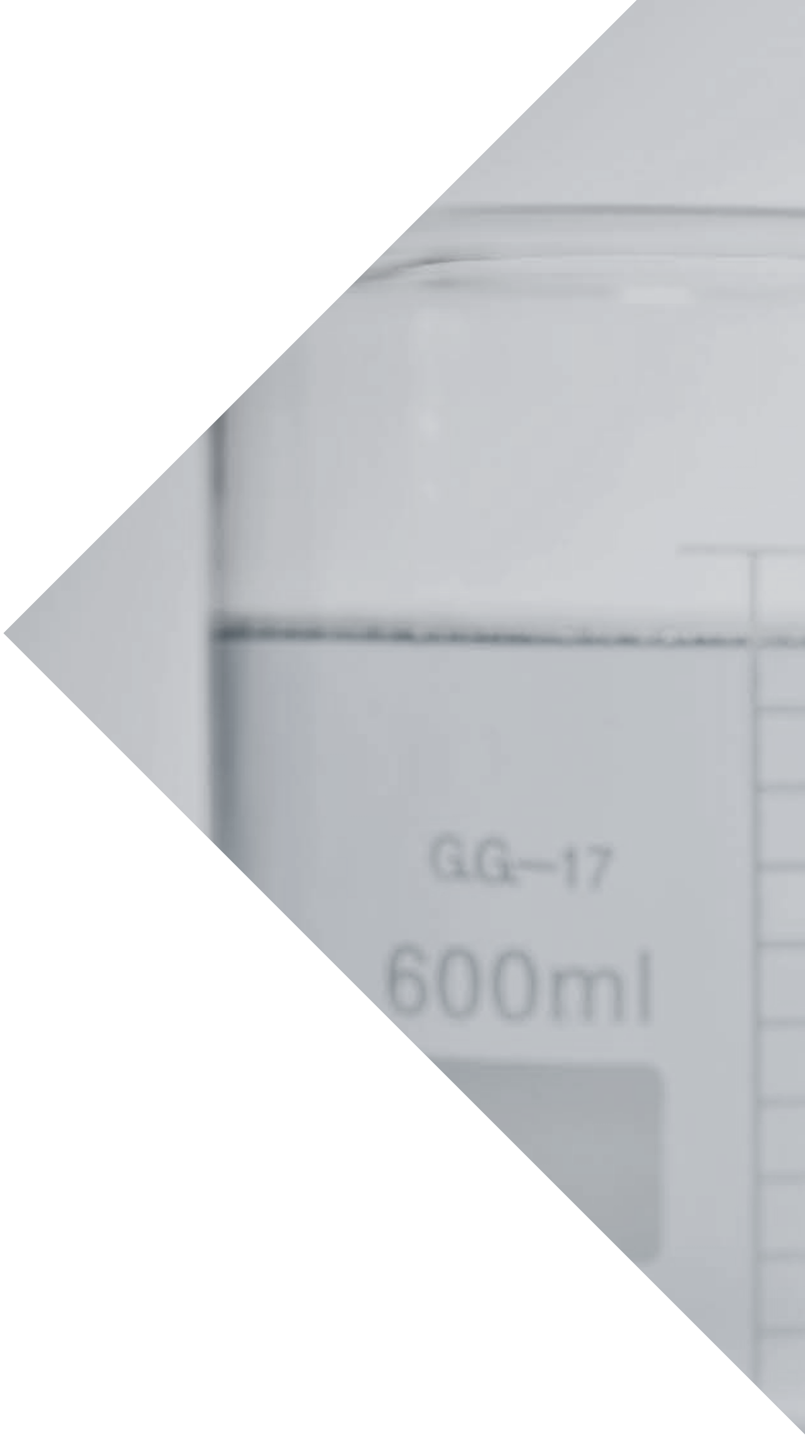

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