

Formulating Water-Based Systems with Propylene-Oxide-Based Glycol Ethers

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To comply with environmental regulations, formulators have reformulated to water-based systems using non-hazardous air pollutants (non-HAP) cosolvents and developed new resin technology. In fully formulated water-based systems, however, changing the solvent system to meet environmental regulations has wide ranging effects on viscosity, surface defects, film shrinkage, adhesion, and durability. Formulators often adjust paint viscosity by balancing the levels of cosolvents, surfactants, and rheology modifiers. Reformulating with non-HAPs solvents such as propylene oxide-based glycol ethers (PG-glycol ethers) helps reduce volatile organic content (VOC) to meet environmental compliance and eliminates HAPs reporting requirements. When replacing ethylene oxide-based glycol ethers (EG-glycol ethers) with their PG-glycol ethers, reformulation seems simple enough, particularly if evaporation rates and solubility parameters are matched. A drop-in replacement, however, requires optimization. This study compares the use of PG-glycol ethers in four architectural latex paints and assesses their effects on rheology, drying, and some key performance attributes.

INTRODUCTION

The primary reason for reformulating a solvent system is to meet environmental compliance in volatile organic content (VOC) reduction and reporting requirements. The development of new resins to produce less toxic coatings formulations can also be explored but it is often costly and requires time. Whether dealing with old or new formulations, all formulators should use non-hazardous air pollutants (non-HAPs) solvents to insure compliance with future regulations and to lower the toxicity of the product. Propylene oxide-based glycol ethers (PG-glycol ethers) are one such example of non-HAPs solvents that can be used in reformulating coatings. In addition, glycol ethers based on propylene oxide can offer improved property development versus their HAPs listed counterparts based on ethylene oxide (EG-glycol ethers).

Replacing currently used EG-glycol ethers with the PG-glycol ethers seems simple enough, particularly if evaporation rates and solubility parameters are matched. Table 1 compiles key physical properties that are important in choos-

ing a suitable coalescent solvent for a waterborne coating.¹ However, changing the solvent package in a coating formulation may also necessitate optimization of the other paint components.

First, the viscosity of paint can be affected by the solvent package depending on the thickener used. Paints thickened with hydroxyethylcellulose (HMHEC) show viscosities that are not too susceptible to changes in the solvent. However, paints thickened with hydrophobically modified thickeners are highly dependent on interactions between the cosolvent and other paint components.² Formulators are cognizant of these effects and often optimize paint viscosity by balancing the levels of cosolvent, surfactants, and rheology modifiers. In the past, many formulators have added diethylene glycol n-butyl ether (DB), a 100% water miscible coalescent, to achieve high shear (ICI) viscosity development. This approach has lost popularity due to the increased VOC requirements, the higher cost of rheology additives, and some water susceptibility issues.

Second, in this industry, each formulation is different, therefore each coating must be evaluated independently. Trapping residual solvent in the cured paint or losing the solvent before film formation is crucial to the performance of the coating. The desirability of the retained

solvent in the film is strongly dependent on the end use. The principal positive effect of solvent entrapment is the increased flexibility of the cured coating and possibly superior cure. Negative effects include slow hardness development, poor blocking resistance, low gloss, and poor water and chemical resistance. Losing solvent prematurely leads to inadequate film formation and poor ultimate performance. If we consider a dried paint film as one which no longer flows, the importance of the viscosity on the drying paint is apparent. Formulated paint viscosity is dependent on the viscosity of the external phase, which is water and anything dissolved in it; the volume of the internal phase, including pigment, resin, and agglomerates; and a packing factor, equal to the internal phase volume at critical pigment volume concentration (CPVC). This relationship has been summarized in the Mooney Equation.³

$$\ln \eta = \ln \eta_e + \frac{K_e V_i}{(1 - \frac{V_i}{f})}$$

where

η_e is the viscosity of the continuous, external phase

K_e is the shape constant = 2.5 for spheres

V_i is the volume fraction of the internal phase, and

f is the packing factor = 0.637 for uniform, monodisperse spheres.

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Table 1—Key Properties of Some Propylene and Ethylene Glycol Ethers as Compared to Water

Glycol Ether	Nomenclature	Evaporation Rate nBuAc=100	% in Water @ 25°C	Flash Point °F	Surface Tension dynes/cm @ 25°C
PM	Propylene glycol monomethyl ether	66.0	100.0	89	27.0
PE	Propylene glycol monoethyl ether	47.0	100.0	92	29.7
PNP	Propylene glycol n-propyl ether	22.0	100.0	119	27.0
PTB	Propylene glycol t-butyl ether	25.0	17.5	113	24.4
PNB	Propylene glycol n-butyl ether	7.0	6.4	138	26.3
DPM	Dipropylene glycol monomethyl ether	2.0	100.0	167	29.0
DPNP	Dipropylene glycol n-propyl ether	1.3	18.0	190	25.3
DPNB	Dipropylene glycol n-butyl ether	0.4	5.0	212	28.8
TPM	Tripropylene glycol monomethyl ether	0.2	100.0	242	30.0
PMA	Propylene glycol methyl acetate	34.0	18.0	114	28.0
PEA	Propylene glycol ethyl acetate	26.0	10.0	129	26.3
DPMA	Dipropylene glycol methyl acetate	1.0	12.0	186	28.3
EM	Ethylene glycol monomethyl ether	53.0	100.0	105	30.8
EMA	Ethylene glycol methyl acetate	35.0	100.0	120	34.0
EB	Ethylene glycol n-butyl ether	6.0	100.0	143	26.6
DB	Diethylene glycol n-butyl ether	0.3	100.0	232	30.0
Water	—	36.0	100.0	None	72.0

Temperature, substrate porosity, film build, and air flow also affect paint drying.⁴ In waterborne systems, both temperature and humidity greatly affect the rate of evaporation of water from the paint. The rate of evaporation of the cosolvent is dependent on the temperature.

Finally, short- and long-term stability of the paint can be affected with a change in solvent. For example, in a waterborne system, the water freezes during freeze/thaw testing. This forces the polymer particles to agglomerate in an irreversible manner. This is more likely to occur with surfactant starved resins. Glycol ethers will suppress the freezing of water and should overcome freeze/thaw problems. Heat-aged testing and pH drifts are good screening tools for changes in stability due to changes in the solvent package.

In this experiment, we hope to give the starting point formulations of four main types of waterborne architectural coatings and to provide an indication of expected effects on paint properties when glycol ethers are used as coalescing solvents. By studying the effect of the coalescent solvent on the rheology, gloss, freeze/thaw, heat stability, dry time, and block resistance in four architectural paint systems, general guidelines can be developed to lead to successful reformulation of other similar coatings systems.

EXPERIMENTAL

Suitable Formulating

In this study, the effect of various coalescents was investigated in waterborne architectural coatings. The four

paints used were: a high scrub polyvinyl acrylic (PVA) interior flat latex, a PVA/acrylic blend interior semi-gloss latex, styrenated acrylic interior/exterior high gloss latex, and an acrylic exterior flat latex.

Master batch formulations were prepared according to *Table 2* with the exception that each batch was made without coalescent or thickener. Preparing a master batch helped minimize experimental variance, as well as shorten the time needed to make the coatings. This master batch was divided into three sub-batches. Each sub-batch was thickened with four dry pounds of one of three classes of hydrophobically modified thickeners: hydrophobically modified alkali swellable emulsion (HASE); hydrophobically modified ethoxylated urethane rheology modifier (HEUR); and hydrophobically modified hydroxy-

Table 2—Masterbatch Formulations of Waterborne Coatings Using Four Architectural Acrylic Latex Paints

Main Components	Function	Interior Flat	Interior Semigloss	Int/Ext High Gloss	Exterior Flat
Grind Base					
(ppw)					
Water	Diluent	350	100	50	40
Colloid 643®	Defoamer	3	2	1.5	2
Propylene glycol	Freeze/thaw	40	65	50	35
Ethylene glycol	Freeze/thaw	—	—	—	25
Tamol® 1124	Dispersant	6	8	7	7
Triton® CF 10	Surfactant	—	4	3	1
14% ammonia	Stabilizer	4	4	4	1.5
Titanium dioxide	Hiding pigment	120	250	200	225
Clay	Extender pigment	200	—	—	50
Carbonate	Extender pigment	150	—	—	—
Attapulgite clay	Extender pigment	35	—	—	—
UF clay	Extender pigment	—	50	—	—
NaK alumino silicate	Extender pigment	—	—	—	150
Letdown					
Acrylic resin	Binder	—	130	500	350
PVA	Binder	265	340	—	—
Colloid 643®	Defoamer	3	2	—	2
Water	Diluent	—	80	115	225
Coalescent	Film former	12	14	25	9.5
PVC	—	48	28	18	45

ethylcellulose (HMHEC) were picked because they are representative examples of this class of thickeners.

The sub-batches were further subdivided into seven paints. A suitable amount of coalescent was added to each. The coalescents selected for this study were: diethylene glycol n-butyl ether (DB); an EG-glycol ether, dipropylene glycol monopropyl ether (DPNP); dipropylene glycol monobutyl ether (DPNB); propylene glycol monopropyl ether (PNP); propylene glycol monobutyl ether (PNB); and dipropylene glycol monomethyl ether (DPM); all PG-glycol ethers,* and 2,2,4-trimethyl-1, 3-pentandiol monoisobutyrate (Texanol®).†

All the formulas were based on a "universal" dispersant to accommodate the three thickener types and to minimize incompatibility between thickener and dispersant class. In three of the four formulas, propylene glycol was used as the freeze/thaw agent, since non-HAPs solvents were preferred. In the exterior flat formula, ethylene glycol was used at the supplier's recommendation. All four formulations were approximately 35% volume solids.

Testing

Viscosity: The viscosity was measured to compare the effect each coalescent has on the final paint formulation. Two methods were selected for determining viscosity. The first, the Stormer viscosity, provided a measure of the low-shear viscosity while ICI cone and plate was used to measure high-shear viscosities. The methods used are as described in ASTM Methods: D 562 Standard Test Method for Consistency of Paints Using the Stormer Viscometer and D 4287 Standard Test Method for High-Shear Viscosity Using the High-Shear (ICI) Cone and Plate Viscometer.⁵

DRY TIME: The rate of evaporation of water is highly dependent on temperature and humidity. For this reason, weather conditions were simulated in a humidity cabinet as described in Table 3 to evaluate drying. To remove any substrate and film build-up effects, the paint dry times were determined on draw-downs done on Leneta charts.

Film drying was measured using a procedure loosely based on ASTM D 1640 Standard Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature.⁵ We

Table 3—Conditions for Drying for Waterborne Paints

Cold & humid	40°F/90% relative humidity
Cold & moderate	40°F/55% relative humidity
Moderate	75°F/45% relative humidity
Hot & humid	90°F/90% relative humidity
Hot & dry	90°F/30% relative humidity

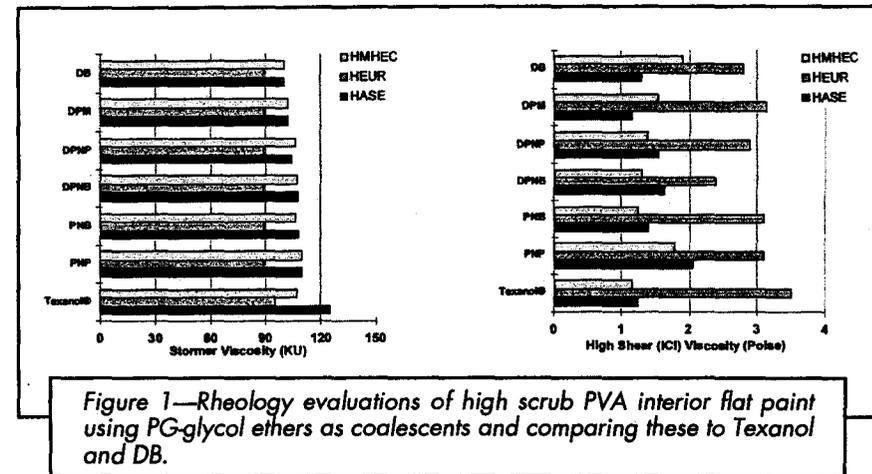


Figure 1—Rheology evaluations of high scrub PVA interior flat paint using PG-glycol ethers as coalescents and comparing these to Texanol and DB.

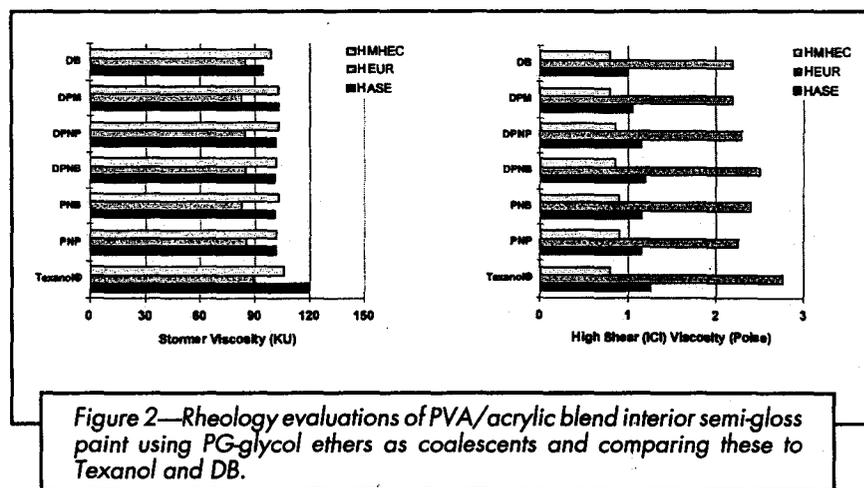


Figure 2—Rheology evaluations of PVA/acrylic blend interior semi-gloss paint using PG-glycol ethers as coalescents and comparing these to Texanol and DB.

evaluated the paints every hour and assigned the following ratings:

Rating	Observation
0,1,2	wet to sticky
3,4,5	dry to touch
6,7,8	dry hard
9	dry through

✓ **Gloss:** Gloss coalescence and degree of agglomeration were examined using the ASTM D 523 Standard Test Method for Specular Gloss for gloss.⁵

STABILITY STUDIES: Freeze/thaw testing with the glycol ethers was designed to look at the cosolvent's ability to depress the water's freezing point. This ability has been measured using ASTM D 2243 Standard Test Method for Freeze-Thaw.⁵

Shelf stability was evaluated using accelerated conditions as described in ASTM D 1840 Standard Test Method for Package Stability of Paint.⁵

FILM PERFORMANCE: Film integrity performance was measured using two tests. The first, ASTM D 2486 Standard Test Method

for Scrub Resistance of Interior Latex Flat Wall Paints, showed the films ability to resist abrasives.⁵ The second method, ASTM D 4946 Standard Test Method for Blocking Resistance of Architectural Paints, provided a measure of the films ability to resist softening.⁵

RESULTS AND DISCUSSION

Rheology

The rheological evaluation using three thickeners for each of the four resins is summarized in Figures 1-4.

Overall, use of the HASE thickener in both the flat and semi-gloss interior formulations with the propyl- and butyl-based PG-glycol ethers offered increased high-shear viscosity over both DB and Texanol (Figures 1 and 2). In the case of the high gloss interior/exterior and the flat exterior, there was no real difference between the solvents (Figures 3 and 4).

*PG-glycol ethers are sold under the Arcosolv® tradename and are a product of Lyondell Chemical Worldwide Inc.

†Texanol® is a registered tradename of Eastman Chemical.

In these same interior formulations, however, there is a significant—25 to 50 KU—Stormer viscosity difference between those containing the solvents DB and Texanol. Formulations containing PG-glycol ethers, on the other hand, developed Stormer viscosity that fell between the formulations using DB and Texanol. With HMHEC thickener, the difference in the range of Stormer viscosity is small, 10 KU units, in the interior paints. The interior flat formulations containing DB HMHEC developed a higher high-shear (ICI) viscosity than the one containing Texanol. One explanation for this behavior is that Texanol often builds low shear viscosity too quickly, possibly lim-

iting the amount of additional thickener that can be added, which falls short of the amount required for high-shear (ICI) viscosity development. Consequently, DB is often recommended as the solvent to use in these formulations. Paints formulated with the PG-glycol ethers and the HMHEC thickener showed high-shear (ICI) viscosities falling between Texanol and DB, with the exception of PNP in the interior flat paint, which was only slightly higher than Texanol. HEUR thickeners are less efficient than the other rheology modifiers for PVA systems. Texanol-based formulations developed the highest Stormer and high shear (ICI) viscosity with this thickener.

Despite the overall trends observed, there are specific examples throughout the experimental series that should be discussed separately. The 25/75 blend of acrylic PVA, with a six percent coalescent level used as the interior semi-gloss formulation, showed the range difference in Stormer viscosity between formulations containing DB and Texanol using the HASE thickener was not as large as in the interior flat coating (Figure 2). The PG-glycol ethers-based paints again fell between these two. There were no significant differences in high-shear (ICI) viscosity between the coalescents. Additionally, when using the HMHEC thickener, the PG-glycol ether containing paints showed low-shear viscosities between those formulations using DB and Texanol. DB containing formulations did not have the advantage of increased high-shear viscosity development in this case. In the HEUR thickened example, the PG-glycol ether paints developed Stormer and high-shear (ICI) viscosities similar to DB-based paints. The Stormer viscosity for the blended PVA/acrylic Texanol formulation was 24 KU higher than DB; this possibly limits the high-shear (ICI) development of the formula.

Only four dry pounds of thickener were used in all the formulations; therefore, the interior / exterior high gloss coating did not have the required viscosity for high gloss paint. However, the data does show viscosity trends with the different solvents (Figure 3). With both HMHEC and HASE thickeners, the glycol ethers-based paints significantly depress the low- and high-shear viscosity relative to Texanol-based paints. HEUR thickeners are recommended in these high gloss systems. The glycol ethers-based and HEUR formulations suppressed low-shear viscosity and, therefore, none showed sufficient high-shear viscosity development. In this case, formulators must balance low- and high-shear development with a blend of glycol ether and Texanol.

Usually exterior paints contain HMHEC and HEUR rheology modifiers. They are based on soft, flexible polymers and only require a five percent coalescent level (Figure 4). The HASE thickened sample showed lower Stormer viscosities for all the glycol ether-based paints as compared to Texanol. On the other hand, the PG-glycol ethers-based paints improved high-shear (ICI) viscosity development versus DB-based paints. With the HMHEC thickener, the PG-glycol ether paints have better Stormer and high-shear (ICI) viscosity than DB-based paints. Additionally, the PG-glycol ethers performed comparably to Texanol-based formulations. Using DPNB as the coalescent afforded the highest high-shear (ICI) viscosity. Also,

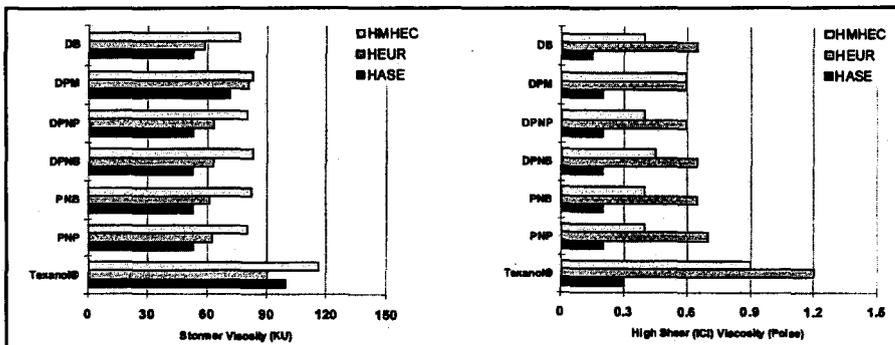


Figure 3—Rheology evaluations of styrenated acrylic interior or exterior high gloss paint using PG-glycol ethers as coalescents and comparing these to Texanol and DB.

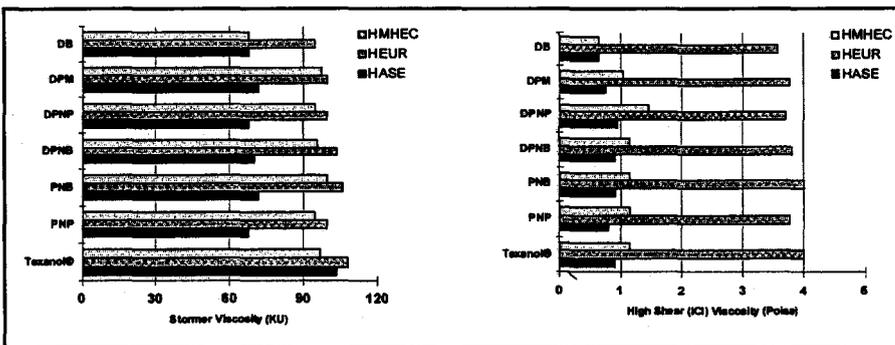


Figure 4—Rheology evaluations of acrylic exterior flat paint using PG-glycol ethers as coalescents and comparing these to Texanol and DB.

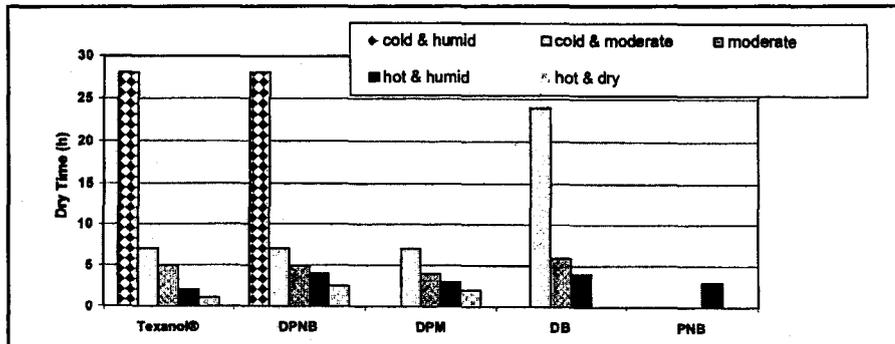


Figure 5—Coalescent effect on dry time of a styrenated acrylic interior/ exterior high gloss paint.

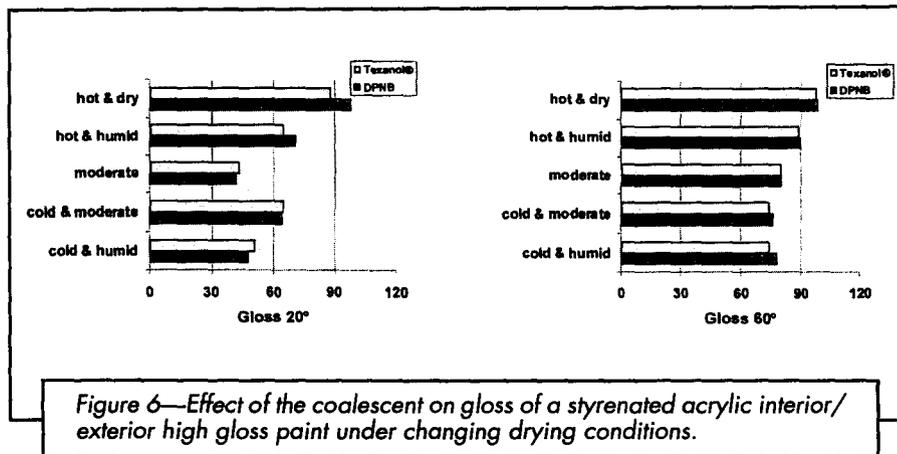


Figure 6—Effect of the coalescent on gloss of a styrenated acrylic interior/exterior high gloss paint under changing drying conditions.

in the HEUR thickened paint, the DPNB coalescent showed viscosity development similar to Texanol.

Film Formation

DRYING UNDER MARGINAL CONDITIONS: The effects of different simulated weather conditions on drying were examined for several coalescents in two exterior latex paint formulations (Figure 5). Five drying conditions were evaluated by noting the time it took the paint to dry (Table 3). The range of coalescents examined had different water miscibility and evaporation rates (Table 1).

Overall, the PG-glycol ethers containing a butyl group show low solubility in water and possess the slowest relative evaporation rates. DB is totally water miscible and slower than the PG-glycol ethers. Texanol is not miscible in water and is the slowest to evaporate.

DRYING OF HIGH GLOSS HEUR THICKENED PAINT: The films' drying time was evaluated using the ASTM Method D 1640 as well as visual inspection of the film for cracking, peeling, and other surface defects. Under cold and humid conditions, the high gloss exterior paints coalesced with Texanol and DPNB while DB, DPM, and PNB failed to form a film within 24 hr dry time. The combination of faster evaporation rates and water miscibility contributed to the failures. Early drying of the paint was fairly consistent for all of the coalescents evaluated.

At lower humidity, only the PNB paint failed to form a film at low temperature. Cracking occurred within the first three hours of drying. The PNB evaporation rate was too rapid. DB and DPNB showed a slight loss of adhesion between four and six hours' dry time, but they both ultimately formed films. Again, early drying of all of the paints was fairly consistent for the coalescents evaluated.

In fact, the evaporation rate of PNB was too fast and failed to form films

under most conditions. The DB failures suggest that it has coupled with the water and has left too quickly to provide adequate film formation. The Texanol coalesced paint had dried-through by the first hour, suggesting a need for the improved "open time," a property attributed to glycol ethers.

DRYING OF EXTERIOR HMHEC THICKENED FLAT PAINT: We repeated the same experiment with the exterior flat formulation. This time only DPNB and Texanol were included. Under all five of the conditions evaluated, the DPNB and Texanol dried the same as in the interior/exterior paint. DB showed poor drying throughout the entire spectrum and was eliminated from the test results.

The most significant observation was during drying under cold and humid conditions. The paints took four days to dry-through. Additionally, the films exhibited a loss of adhesion during this time, indicating a susceptibility to rain and dew. One explanation is that the higher pigment volume concentration tends to increase the paint porosity. The exterior paint was the only paint that contained both EG-glycol ethers and PG-glycol ethers in the same formulation. It is well known that EG-glycol ethers are very hygroscopic.⁶ With the increased porosity and high humidity and low temperature conditions, the water in the ambient can easily migrate into the unformed film, saturate it, and hinder the drying process.

Overall, the experiment was designed to highlight

the differences in drying under several conditions. In both formulations, we observed that substituting DB with DPNB improves film formation of the paints. DPNB also offers increased open time for the high gloss paint.

PERFORMANCE

Gloss under Marginal Drying Conditions

We evaluated the 20° and 60° gloss of the HEUR thickened high gloss paints after they reached dry-through under the various drying conditions (Figure 6). These paints were evaluated for 3 mil drawdowns on Leneta charts. A correlation between gloss and drying condition was observed. When dried under hot/dry conditions, DPNB-based formulations showed approximately 100% gloss retention, while those based on Texanol lost about 12% of the 20° gloss. This may be a result of snap drying of the Texanol-based paint. Under hot and humid conditions, the 20° gloss for the DPNB-based paint fell 30% and both coalescents showed 60° gloss loss of 10%. Both Texanol and DPNB were at 35% of their 20° gloss, and 25% of their 60° gloss, under cold and moderate conditions. Finally, under cold and humid conditions, both DPNB and Texanol formulations were at 50% of their 20° gloss and 75% of their 60° gloss.

Shelf Stability, Freeze/Thaw Resistance, Scrub Resistance

The formulated paints were evaluated for shelf stability, freeze/thaw resistance, block resistance, and scrub resistance using conventional test meth-

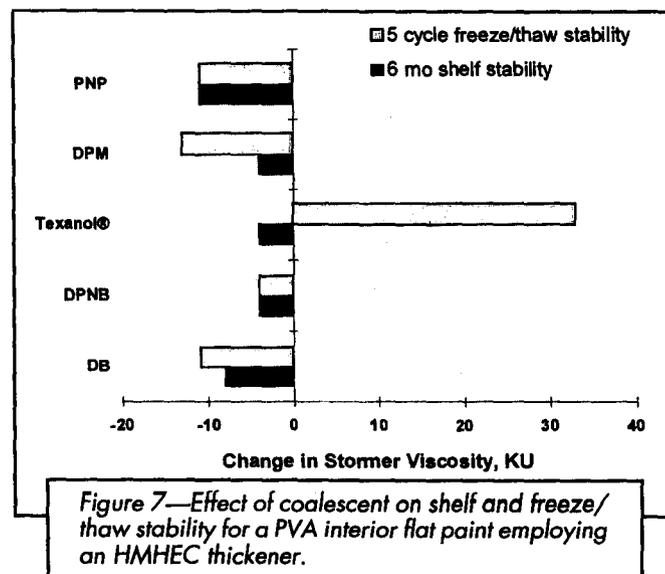
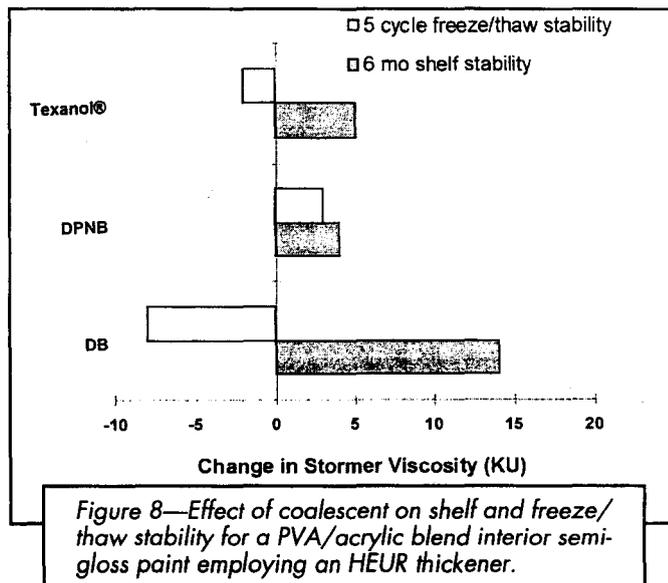


Figure 7—Effect of coalescent on shelf and freeze/thaw stability for a PVA interior flat paint employing an HMHEC thickener.



ods. In many tests, the formulations behaved equally and were not reported. There were, however, results that clearly separated some PG-glycol ether coalescents from the DB and Texanol benchmarks.

In interior flat paint using HMHEC, the six-month shelf stability for DPNB-, DPM-, and Texanol-based paints was excellent. Using DB as the coalescent was acceptable while the PNP based formulation was only marginal (Figure 7). The five-cycle freeze/thaw stability was excellent with DPNB as the coalescent and marginal with DPM-, PNP-, and DB-based formulations. The Texanol formulation failed the freeze/thaw stability test. The abrasive scrub results for paints based on DB, DPNB, and Texanol were equivalent while those based on DPM and PNP were only slightly, 20%, lower.

In the interior semi-gloss paint, the DB-based formulation had poor shelf and freeze/thaw stability, while both the DPNB and Texanol formulation showed excellent shelf and freeze/thaw stability (Figure 8). Texanol-based paint produced superior scrubs to formulations based on DB and DPNB, these being equivalent. The DPNB-based formulation, however, significantly improved the overnight, ambient block resistance rating to eight versus six for DB and five for Texanol. Oven block resistance was equivalent for all formulations.

CONCLUSIONS

In all four formulations evaluated, the low shear, Storer viscosities of the PG-glycol ethers were between DB and Texanol regardless of the thixotrope used. Texanol-based formulations showed the higher Storer viscosity while DB was at the low end. In comparison, the PG-glycol ethers provided enhancement in KU development with the propyl and butyl ethers contributing the most to this enhancement. PG-glycol ethers formulated paints also presented improved high-shear viscosity over those using Texanol, when either HMHEC or HASE was used in the formulation. Likewise, the same behavior is observed in the exterior paints. In this case, a combination of PG-glycol ethers and Texanol would be desirable.

Drying time in the exterior formulations that were evaluated showed improvement with DPNB. PNB showed cracking and failure to dry under most weather conditions. DPM presented a midpoint between DB and DPNB. DB failed to dry under cold and humid and hot and humid conditions. Only two formulations dried under extreme cold and humid conditions; DPNB dried not unlike Texanol. DPNB performs similarly to Texanol under cold conditions and offered a slight improvement in gloss under hot conditions. Again, the widest

formulating latitude could be a combination of a PG-glycol ether and Texanol.

Additionally, the PG-glycols showed good stability under both freeze/thaw conditions and shelf stability testing. They demonstrated good block resistance. Texanol failed the freeze/thaw test, again suggesting a blended system may enhance the long-term stability of paints, which are formulated with this coalescent.

PG-glycol ethers offer an attractive balance of performance. This was especially true for interior flat formulas based on high scrub PVA. With further optimization these solvents present an alternative to the formulator. This study provides a starting place for launching a complete investigation of film properties to assure paint performance when using PG-glycol ethers.

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