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FORMULATING POLYURETHANE DISPERSIONS

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ABSTRACT

Polyurethane dispersions are being used increasingly in industrial applications. Because of the high cost of these dispersions they are being employed where the performance achieved with waterborne acrylic, alkyd, polyester and epoxy resins is not adequate, or they are being used in combination with other resins to improve certain properties. I will discuss the problems associated in formulating polyurethane dispersions, how these systems can be pigmented, and crosslinked with either melamine or polyisocyanate crosslinker and how to catalyze these formulations. In addition I plan to discuss how to combine these polyurethane dispersions with other polymer systems.

INTRODUCTION

Solvent-borne polyurethane polyols are used in many industrial applications where high abrasion resistance, elastomeric properties, and high extensibility at low temperature are the performance requirements.¹⁻¹⁴ Most of the solvent-borne PUR polyol coatings are relatively high in solvent content and do not qualify in low VOC applications. For this reason, there is an increased interest in water-borne crosslinked PUR coatings.

Polyurethane dispersions are being synthesized by different methods and processes compared to acrylic or other vinyl dispersion polymers. Most of the acrylic and vinyl dispersions are produced by an emulsion polymerization process in the presence of an external surfactant or colloidal stabilizer. Most of the polyurethane dispersions on the other hand are produced by a prepolymer process¹⁵ which involves the preparation of a low molecular weight isocyanate terminated ionomer, either in bulk or in a solvent, dispersing this ionomer in water followed by chain extension. To be able to chain extend the polymer in water, the low molecular weight prepolymer is prepared from a diol, an ionic monomer and an excess of diisocyanate. After dispersion of the ionomer in water, chain extension takes place by reaction of the isocyanate end groups with water and reaction of the resulting amine with more isocyanate, or by addition of a di functional primary amine to the dispersed polyurethane. Most acrylic and vinyl polymers are produced with a low amount of ionic co-monomer.

Polymerization of the dispersion is carried out in most systems under acidic pH condition and the ionic groups on the polymer are not ionized. After polymerization the dispersions are neutralized and ionized and contribute to improved stability of the dispersion polymer to freeze thaw, to pigmentation and shear. In a polyurethane prepolymer the ionic groups are placed on the backbone in a random placement. During dispersion and neutralization these ionic groups are solvated by the water and orient themselves as much as possible onto the outside of the polymer micelle. Because of restriction in mobility this is an incomplete process. Many ionic groups can be buried in the polymer particle after the dispersion process¹⁶. We also prepared low molecular weight polyurethane dispersions by a non-isocyanate process which utilized a urethane diol which was transesterified with a polyester resin and functionalized with carboxyl groups in a second reaction step¹⁷. These carboxyl and hydroxyl functional polyester-urethane resins have a MW of between 1000 to 5000. These resins can be either used in a solvent borne coating or can be dispersed in water in the presence of an amine.

In the acrylic polymerization process most if not all of the ionic groups migrate to the surface of the particle, making the center hydrophobic and essentially devoid of any functional groups. Typical polymer dispersions are characterized by a minimum film formation temperature (MMFT). This is the lowest temperature at which the dispersion polymer particles can coalesce and form a continuous film. For most acrylic dispersions this MMFT is close to the glass transition temperature (Tg) or slightly below the Tg. Coalescence requires the deformation of the polymer particles and inter diffusion of the polymer chains. The MMFT can be lowered in a dispersion by the addition of a solvent which acts as a temporary plasticizer for the polymer. In addition film formation can be lowered. Film formation of colloidal waterborne resins is less dependent on the glass transition temperature of the polymer and these polymers can form a film substantially below the Tg of the polymer. In this case the section of the polymer containing ionic groups are being plasticized by water during film formation. Most so called "water soluble" polymers fall into this category.

Most polyurethane dispersion fall into the category of colloidal dispersions or ionomer. The ionic layer on the particle surface and also some hydrophilic groups within the particle are plasticized by the water, therefore film formation can take place above the glass transition temperature (Tg) of the polymer. Polyurethane dispersions offer a unique combination of properties, flexibility, toughness, high abrasion resistance and if aliphatic excellent light stability. Compared to acrylic polymers, however, polyurethanes suffer from poorer water, chemical resistance and hydrolytic stability. For most industrial applications an polyurethane dispersions can be improved by crosslinking of the polymer. Some of the polyurethane properties, however, can be incorporated into other polymers by either blending or by co-reaction.

EXPERIMENTAL

MATERIALS

The low molecular weight hydroxyl and carboxyl functional polyurethane dispersions (PUD) were prepared by a non-isocyanate process. The higher molecular

weight dispersions were prepared by a conventional isocyanate prepolymer process or commercial materials were used. The polyester urethanes were dispersed in water after addition of an amine. The characteristics of a low MW PU dispersions used in the pigment dispersion studies and in some of the crosslinking studies is shown in Table 1 and 2.

PIGMENT DISPERSIONS

Both organic and inorganic pigments were dispersed in the PUD XM-2311¹⁸. The pigment is dispersed in the PUD additional water or amine is added and the paste is dispersed. The carbon black is ground in a steel ball mill for 24-72 hours depending on the degree of jetness and dispersion desired. The organic pigments were dispersed first on a high speed dissolver and then ground in a porcelain or ceramic ball mill. The titanium dioxide was dispersed in a high speed mixer for 15-20 minutes. A Hegman fineness of 7+ is easily achieved.

CROSSLINKING OF PUD

Crosslinking in this study was carried out with a water dispersible polyisocyanate crosslinker¹⁹. This crosslinker was modified with nonionic sites to improve dispersibility²⁰ (Table 3). The crosslinked films were tested for hardness (Pendulum), solvent resistance (MEK rub) and also for gloss. Film properties as a function of cure time and also pot life of the formulation was investigated. The formulations were studied with and without any additional catalyst.

RESULTS AND DISCUSSION

PIGMENT DISPERSION

In testing the quality of the dispersion it is important to test the dispersion after letdown with a binder. Particle size analysis of the PUD XM-2311 pigment dispersion show a particle size of about 1 micron. The pigment is dispersed within the hydrophobic phase of the polyurethane dispersion. This behavior can explain why pigment dispersions with XM-2311 can be formulated at a high solids. Picture 1 shows a pigment dispersion prepared according to Table 4 with a pigment to binder ratio of 1/1 using a Irgazin DPP BO red pigment and a grinding time of 16 hours on a pebble mill and let down with a PUD dispersion. The same pigment dispersion diluted with water shows particles of about 1 micron, dilution with a solvent shows agglomeration of particles (Picture 2). Besides Irgazin red, which is a rather large particle size pigment a wide selection of organic and inorganic pigments were dispersed. The results of this dispersion work are shown in Table 5.

CROSSLINKING WITH POLYISOCYANATE

In this study we investigated the crosslinking reaction and catalysis of a hydroxyl/carboxyl functional PUD²¹ with a water dispersible polyisocyanate¹⁹. One of the major problems in crosslinking of hydroxyl functional polymers in water is the side reaction between the isocyanate groups and water leading to the formation of polyureas and a decrease in crosslinking density. Almost all waterborne formulations of polyols with polyisocyanates are significantly overdosed with polyisocyanate, often a 100 % excess of polyisocyanate is used. In this study various commercially available metal carboxylate and chelate catalysts were compared to dibutyltin dilaurate (DBTDL) and their effect on crosslinking versus polyurea formation was monitored. We also investigated the effect of catalyst on the useful pot life of a coating formulation.

To illustrate the reaction of water with a polyisocyanate we added both a DBTDL and the XC-4205 metal chelate catalyst to the polyisocyanate and also added 2 % of water to the formulation. The results are shown in Picture 3. The DBTDL containing formulation starts to foam after a short incubation period, the XC-4205 containing formulation shows only a small amount of gassing.

A formulation containing a carboxyl and hydroxyl functional PUD dispersion and a water dispersible polyisocyanate crosslinker (Table 6) was catalyzed with DBTDL, K-348 a bismuth octoate catalyst, XC-5218 a chelated aluminum and XC-4205 a chelated zirconium catalyst. This formulation is an elastomeric coating for soft feel application. A catalyst level of 0.01 % metal (tin and K-348), 0.0012 % (XC-4205) and 0.078 % (XC-5218) metal based on total binder was used. The catalysts was diluted with the dimethoxy dipropyleneglycol solvent before addition to the PUD. Pendulum hardness of this formulation applied immediately after mixing and 2 hours after mixing is shown in Figures 1 and 2. Figure 3 gives the changes in gloss (60°) after aging the formulations from 0 to 7 hours before application. Figure 4 gives the MEK resistance of formulations again after a 0 to 7 hour aging time of the formulation. In Figure 1 it can be seen that all catalysts appreciable increase the hardness, this includes the DBTDL. On the other hand after only 2 hours of potlife the DBTDL catalyzed formulation does not cure well, as shown in Figure 2, 3, and 4. For all the other catalysts the effective potlife without any appreciable changes in properties is about 3 hours. The formulation containing no external catalyst has a slightly longer potlife of about 4 hours, but the film properties never approach the characteristics of the catalyzed formulations. The effect of potlife on gloss of a DBTL and XC-4205 catalyzed formulation is also shown in Picture 4.

The performance characteristics of a room temperature and forced air-dried coating formulation crosslinked with a water dispersible PUD is shown in Table 7. Characteristic for this dispersion are elastomeric behavior and complete recovery on deformation. Pencil hardness is 2H of the crosslinked formulation, but König Pendulum is below 100.

TABLE 8 gives the results of modification of an acrylic dispersion crosslinked with a water dispersible isocyanate with a low molecular weight PUD. The system was formulated with two different levels of OH/NCO ratio, 1/1 and 1/1.6. The PUD modification in this formulation raises the gloss, solvent resistance and increases the impact resistance at some sacrifice in Pendulum hardness. The VOC of all formulations is between 60 to 40 g/l. Because of the use of the high MW dispersion overall NCO

demand is low. The acrylic dispersion also permits very fast drying time, this is initially physical drying, later followed by crosslinking. The modification with the PUD decrease drying time at no sacrifice in potlife. Both the XM-4316 and the acrylic dispersion alone blended with the water dispersible polyisocyanate give a useful potlife in excess of 3 hours. The formulations gelled after approximately 6 hours.

CROSSLINKING WITH MELAMINE RESINS

A black single coat formulation utilizing PUD XM-2311 as a pigment grinding agent, PUD XM-4316 as a backbone resin and HMMM as a crosslinker is shown in Table 9. To prepare the pigment paste the XM-2311 polyurethane dispersant resin is blended with water and dimethyl ethanolamine and the carbon black is added. This blend is premixed and the blend of K-SPERSE²² with dimethyl ethanolamine is added. The pH should be above 9.2. The entire mixture is dispersed in a steel ball mill to Hegman NS 8 rating. Grinding can take 48-72 hours depending on the jetness desired. After grinding in the steel ball mill add the additional PUD XM-2311, XM-4316 and water to the ball mill, run for an additional 30 minutes. This pigment concentrate is stable. After longer aging addition of small amounts of amine for pH adjustment might be required. The pigment paste is let down under agitation with the remainder of PUD XM-4316, water, melamine crosslinker, silicone surfactant and catalyst. The final pH of the formulation should be between 7.8 to 8.3. This formulation has excellent adhesion to iron phosphated steel panels and give 350 hours salt spray without any major creepage from the scribe line. Salt spray performance of a PUD dispersion prepared by a conventional isocyanate process in the same formulation gives significant blistering only after 100 hours exposure.

CONCLUSIONS

Low molecular weight waterborne PUD's have shown excellent pigment dispersion properties and can be used effectively to disperse a wide range of both inorganic and organic pigments. In industrial applications PUD's crosslinked with ether polyisocyanate and melamine crosslinker give excellent properties and can also be used to modify other polymer systems.

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TABLE 1 CHARACTERISTICS OF PU XM-2311

APPEARANCE	Clear or hazy dispersion
PARTICLE SIZE, nm	10-20
NONVOLATILE, 60 min. 110°C, %	40
SOLVENT	water
NEUTRALIZING AMINE	TEA/DIOPA
pH	8.8
HYDROXYL NUMBER (SOLIDS)	33
ACID NUMBER (SOLIDS)	60
AMINE CONTENT	0.375 MEQ/g of dispersion
EQUIVALENT WEIGHT (SOLIDS)	603
EQUIVALENT WEIGHT (SOLUTION)	1493
VISCOSITY, Brookfield, SP16, 60 RPM, 25°C, cp.	500
SPECIFIC GRAVITY, g/cc	1.054
FLASH POINT, CLOSED CUP, °F	130
URETHANE CONTENT, MOL/1000	1.5
MOLECULAR WEIGHT, approx.	1500

TABLE 2 CHARACTERISTICS OF PU XM-4316

APPEARANCE	Bluish dispersion
NONVOLATILE, 60 min. 110°C, %	40
SOLVENT	water
NEUTRALIZING AMINE	n-Methyl morpholine
pH	8.0
HYDROXYL NUMBER (SOLIDS)	60
ACID NUMBER (SOLIDS)	40
AMINE CONTENT	0.28 MEQ/g of dispersion
EQUIVALENT WEIGHT (SOLIDS)	560
EQUIVALENT WEIGHT (SOLUTION)	1700
VISCOSITY, Brookfield, SP16, 60 RPM, 25°C, cp.	500
SPECIFIC GRAVITY, g/cc	1.06
FLASH POINT, CLOSED CUP, °F	>200
URETHANE CONTENT, MOL/1000	0.6
MOLECULAR WEIGHT, approx.	3000

TABLE 3 CHARACTERISTICS OF WATER-DISPERSIBLE
POLYISOCYANATE (XP-7007)

Viscosity, 25°C, cps	3000
NCO weight, %	19-19.5

TABLE 4 GRIND FORMULATION WITH PUD XM-2311

GRIND FORMULAS.....	A	B	C	D
Pigment / Binder	1	1.2	1.4	2.46
PIGMENT LOADING	0.25	0.3	0.35	0.45
PUD XM-2311 (43.50%)	57.5	57.5	57.5	42
Irgazin DPP BO RED	25.0	30.0	35.0	45
Water	17.5	12.5	7.5	13
Total	100	100	100	100
Nonvolatile, %	49.2	54.2	59.2	62.6

TABLE 5 GRIND FORMULATION WITH PUD XM-2311

Material	Description	Carbon Black	Phthalo Blue	Perylene Maroon	Phthalo-cyanine Green	Diarylide Yellow	Opaque Orange
K-FLEX XM-2311	PU-dispersion	36.2	52.0	34.3	37.0	36.3	47.6
Pigment		15.00	25.0	35.00	48.8	39.8	41.7
Dimethyl ethanol amine	Neutralizer	3.7					
Water	Diluent	45.2	23.0	30.7	14.2	23.9	10.7
Total		100.0	100.0	100.0	100.0	100.0	100.0
Formulation Characteristics							
Resin Solid, Weight, %		15.2	21.8	14.4	15.6	15.3	20.0
Pigment to binder ratio		1/1	1/1	2.5/1	3/1	2.5/1	2/1

TABLE 6 PUD CROSSLINKED WITH POLYISOCYANATE

PUD XM-4316 POLYMER	28.1
PUD XM-4316 WATER	42.1
SOLVENT DIMETHOXYDIPROPYLENEGLYCOL	1.8
WATER	12.2
BAYHYDUR XP-7007	15.8
TOTAL	100.0

TABLE 7 PUD CLEARCOAT FORMULATION

COMPONENT	DESCRIPTION	% BY WEIGHT
COMPONENT A		
K-FLEX XM-4316	Polyurethane dispersion	70.2
Water	Diluent	12.2
Proglyde DMM	Solvent	1.8
COMPONENT B		
Bayhydur XP-7007	Water-dispersible isocyanate	15.8
Total		100.0
Formulation properties next page.		

Table 7 continued.

FORMULATION CHARACTERISTICS

NCO / OH, ratio		1.7 / 1.0
Nonvolatile, %		47.4
Theoretical VOC, g/L, (lb./gallon)		106, (0.88)
pH Component A (as is)		7.5
Brookfield Viscosity, (25°C), cPs spindle #16, 60 rpm		1500
Dry Times (hours)	set to touch	0.25
	surface dry	2.25
	through-dry	5.75
FILM PROPERTIES		
Substrate, iron phosphated steel		
Cure Schedule, temperature, time	20-25°C, 2 weeks	80 °C, 30 minutes
Dry Film Thickness, mil, (micron)	1.0, (25)	1.0, (25)
Pencil Hardness	2H	2H
König Hardness, (seconds)	41	70
MEK Double Rubs	90	160
Impact Resistance (in.lb)		
Direct, Reverse	>160, >160	>160, >160
Gloss, 60°, 20°, %	>90, >90	>90, >90

TABLE 8 ACRYLIC DISPERSION MODIFIED WITH PUD ISOCYANATE CROSSLINKED

	XM-4316	CONTROL	XM-4316	CONTROL
NCO/OH RATIO	1.1	1.1	1.6	1.6
ACRYLIC/PU-DISPERSION	80/20	100/0	80/20	100/0
Component A				
Acrylic Emulsion E-3275	71.38	87.87	69.31	84.73
PUD XM-4316	17.40	0.00	16.89	0.00
Sodium Nitrite (5% aqueous)	0.00	2.42	0.00	2.33
Proglyde DMM	0.89	0.84	1.23	0.81
Flow and Leveling Agent	0.57	0.78	0.07	0.92
Dimethyl ethanolamine	0.73	0.54	0.00	0.52
Component B				
Bayhydur XP-7063	9.03	7.56	12.50	10.69
TOTAL	100.00	100.00	100.00	100.00
Formulation Characteristics				
Nonvolatile, theoretical, %	46.3	44.5	48.7	46.3
Theoretical VOC, g/L, (lb./gallon)	60, (0.5)	39, (0.33)	48, (0.4)	36, (0.3)

Table 8 continued				
Formulation Properties, Air-Dry 20-25°C, 2 weeks				
Substrate: Bonderite 1000 cold rolled steel				
Film thickness, micron (mil.), 32 ± 2 (1.3 ± 0.1)				
DRY TIME (minutes)				
Set to touch			4.5	14
Surface dry			12	25
Through dry			18	40
Formulation Properties, Air-Dry 20-25°C, 2 weeks				
Gloss 60°, %	95	93	96	84
20°, %	69	69	74	54
Koenig Hardness (sec)	69	105	108	120
Pencil Hardness	H-2H	H-2H	2H-3H	3H
Knoop Hardness	2.4	3.9	5.5	8.2
MEK Double Rubs	80	30	120	80
Impact, Direct, inch.lb.	>160	>160	>160	>160
Impact, Reverse, inch.lb.	>160	150	>160	130
Formulation Properties, Bake 80°C 20 min. Post-cure 20-25°C, 2 weeks				
Substrate: Bonderite 1000 cold rolled steel				
Film thickness, micron (mil.), 32 ± 2 (1.3 ± 0.1)				
	XM-4316	control	XM-4316	control
NCO/OH RATIO	1.1	1.1	1.6	1.6
Gloss 60°, %	96	99	100	94
20°, %	66	79	80	67
Koenig Hardness (sec)	89	114	134	132
Pencil Hardness	2H	4H	3H	4H
Knoop Hardness	3.7	4.9	7	9.7
MEK Double Rubs	120	50	135	105
Impact, Forward, inch x lb.	>160	160	>160	80
Impact, Reverse, inch x lb.	160	150	>160	30

Table 9 PUD CROSSLINKED WITH HMMM BLACK SINGLE COAT		
COMPONENT	DESCRIPTION	% BY WEIGHT
Pigment Grind Paste, Part 1		
K-FLEX XM-2311	Pigment dispersing resin	2.40
Water, deionized	Diluent	2.95
Dimethylethanolamine (1)	Amine neutralizer	0.15
Monarch 1300	Carbon black pigment	1.00
K-SPERSE® 152 MS	Dispersant	0.07
Dimethylethanolamine (2)	Amine neutralizer	0.10
Pigment Paste, Part 2		
K-FLEX XM-2311	Pigment dispersing resin	1.20
K-FLEX XM-4316	Polyester-urethane dispersion	1.20
Water, deionized	Diluent	1.10
Total		10.17

Table 9 continued

Pigment Paste	Pigment concentrate	10.17
K-FLEX XM-4316	Polyurethane dispersion	63.95
Water deionized	Diluent	6.20
Resimene® 747	Amino crosslinker (HMMM)	9.55
NACURE® 2547	Blocked acid catalyst	0.13
Silwet L7604 / L7605 (1:1) 25% in H ₂ O	Flow and leveling agent	0.40
Water, viscosity adjustment	Diluent	9.60
Total		100.00

FORMULATION CHARACTERISTICS

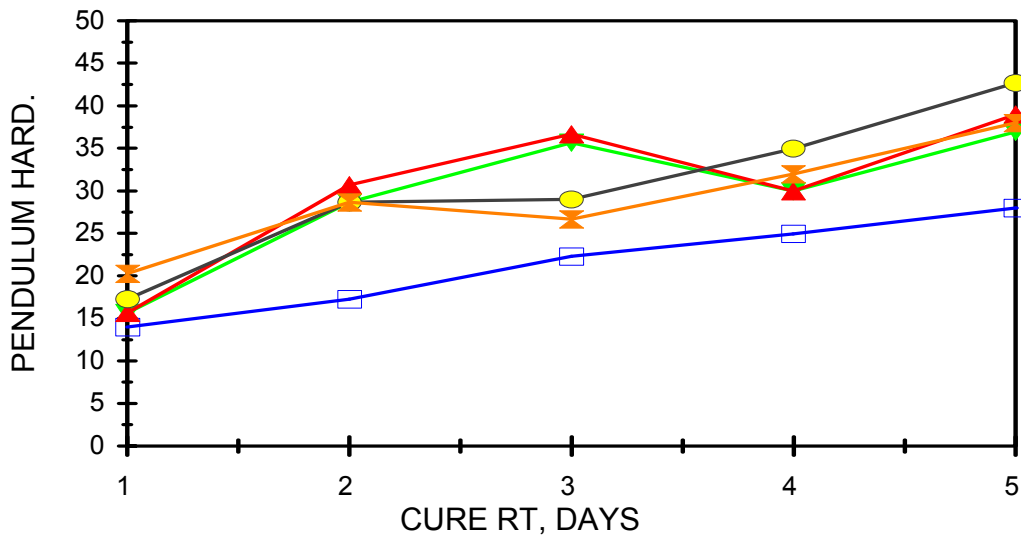
Resin/HMMM, ratio	75/25
Nonvolatile, , calculated, %	39.0
Theoretical VOC, g/L, (lb./gallon)	145, (1.23)
pH	7.8-8.3
Brookfield Viscosity, 25°C, spindle #16, 60 rpm, cp.	300-500

FILM PROPERTIES Substrate, aluminum

Cure Schedule, temperature, time	150°C, 15 minutes
Dry Film Thickness, mil, (micron)	1.0, (25)
Gloss, 60°, 20°, %	91, 81

Figure 1

**WATERBORNE TWO COMPONENT
POTLIFE 0 HOURS**



■ NO CAT
 ▼ XC-5218
 ▲ K-348
 ● XC-4205
 ✕ DBTDL

Figure 2

WATERBORNE TWO COMPONENT POTLIFE 2 HOURS

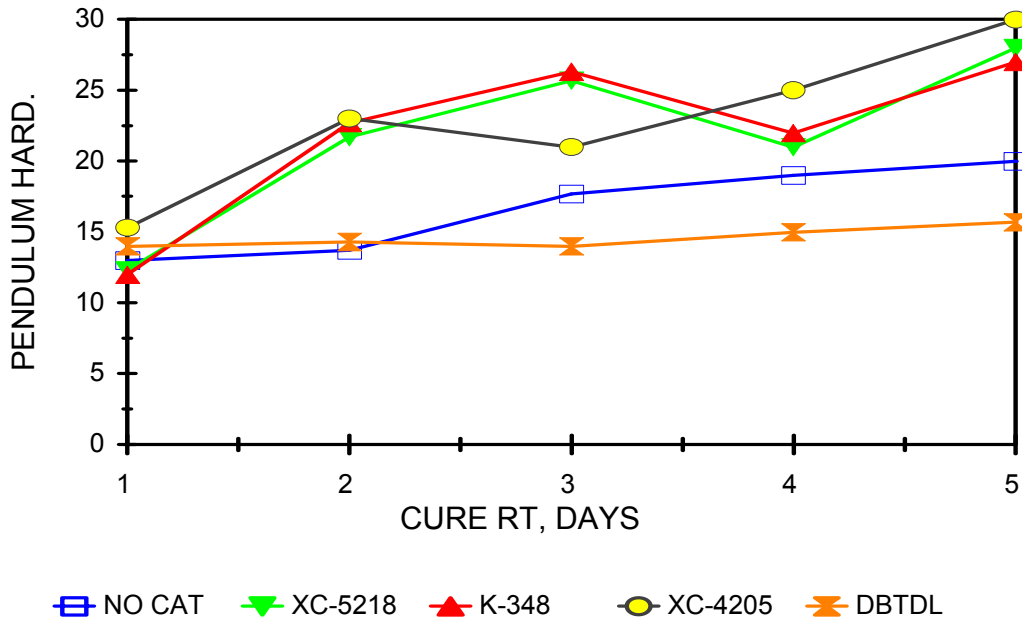


Figure 3

WATERBORNE TWO COMPONENT POTLIFE

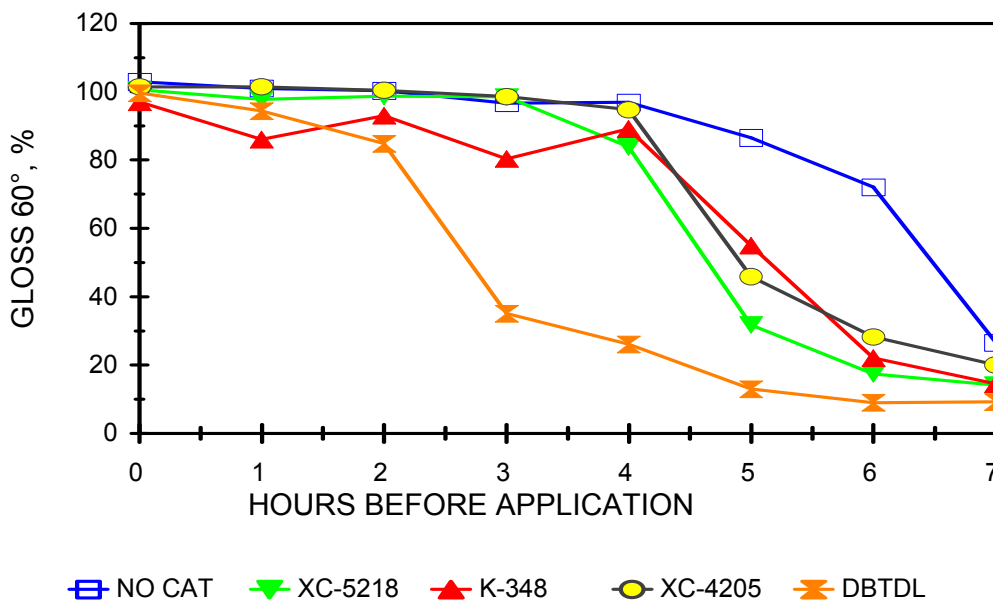
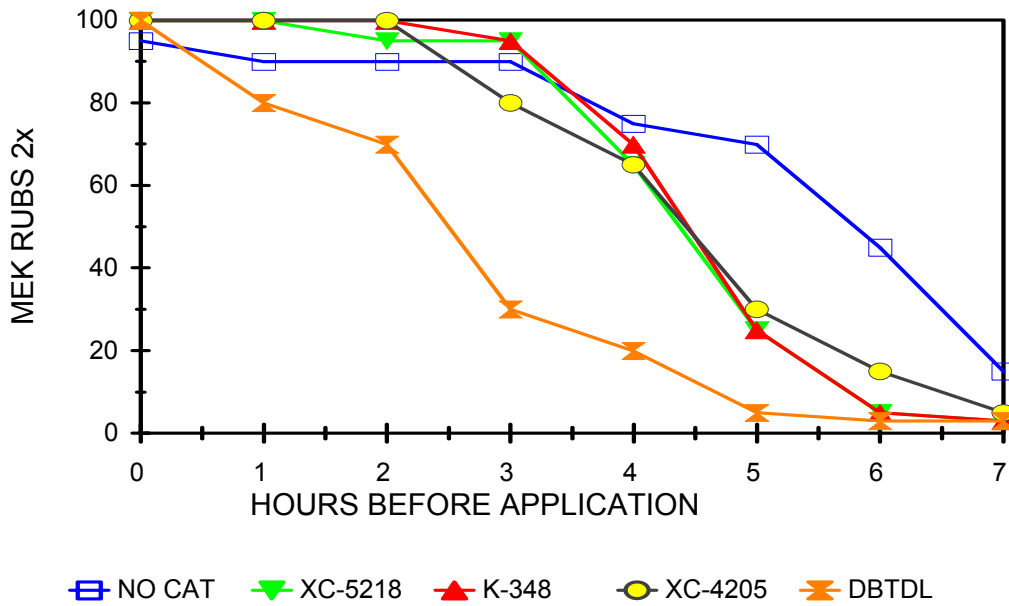


Figure 4

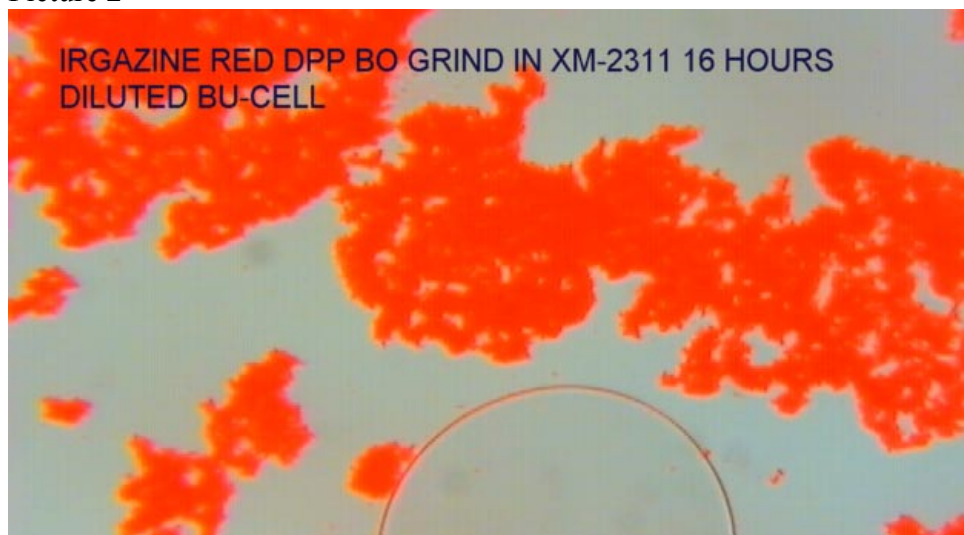
WATERBORNE TWO COMPONENT POTLIFE



Picture 1



Picture 2



Picture 3 Effect of Catalyst on Water -NCO reaction



Picture 4 Effect of Potlife of Two Component Waterborne/polyisocyanate coating on gloss

