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Comparison of Thixotropes in Coatings Formulations Comprising Tertiary-Butyl Acetate (TBAc™)

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Introduction:

There are many issues facing the coatings industry today. One of the most critical is the reduction in VOC's. While acetone and methyl acetate are VOC-exempt solvents, they have very low flash points and evaporate far more quickly than many formulators prefer; see Table 1. Some formulators are turning to OXSOL 100, paracholorbenzotriflouride (PCBTF). But this solvent is costly and has perceived health concerns. Tertiary-Butyl Acetate (TBAc) has recently been listed by many state and local air quality boards as a VOC-exempt solvent. With a more reasonable flash point and evaporation rate, TBAc represents another tool available to coatings formulators. However, traditional rheology modifiers may not be optimized for these new low-VOC formulations. The results of this evaluation show that Garamite® 1958 is a much more efficient thixotrope than traditional organoclays or fumed silica in several low-VOC coatings formulations.

Table 1: Properties of Select Solvents

	TBAc	Acetone	Methyl Acetate	PCBTF
Flash Point	4°C	< -20°C	-13°C	43°C
Evaporation Rate*	2.8	7.7	5.3	0.9

* Butyl Acetate Evaporation Rate = 1

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Experimental and Results:

A. Medium Oil Alkyd Formulations

Volume	Material	Weight
28.81	MOA 11-070	218.98
3.78	Mineral Spirits or TBAc	27.37
0.47	Propylene Carbonate	4.65
0.96	Thixotrope	13.69
	HSD 10 min. and add:	
0.68	Raybo 57HS	5.47
	Mix 1 min. and add:	
7.56	TiO2 CR-800	260.04
	HSD 15 min and add:	
44.30	MOA 11-070	336.68
11.34	Mineral Spirits or TBAc	82.12
0.37	Cobalt 6%	2.74
0.49	Zirconium 6%	3.56
0.88	Calcium 5%	6.84
0.36	Anti-Skinning Agent	2.74
100.00		**964.88**

In a standard medium oil alkyd formulation, TBAc was substituted for all of the free Mineral Spirits. Since TBAc has now been listed as a VOC-exempt solvent, this substitution reduced the coatings VOC from 3.95 lbs/gal to 3.43 lbs/gal. The results of the above formulations are listed in Table 2 and Table 3.

Table 2: Viscosities of Medium Oil Alkyd Formulations

			Brookfield RVT (cps)		
Sample ID	Thixotrope	Solvent	1 rpm	10 rpm	100 rpm
KTO144093A	Claytone 40	Mineral Spirits	9200	2840	1368
KTO144093B	Claytone 40	TBAc	8200	2020	748
KTO144093C	Garamite 1958	TBAc	10200	2060	610
KTO144093D	Garamite LS	TBAc	7400	1720	614
KTO144093E	No Thix/Control	TBAc	800	360	242

As can be seen in Table 2, Garamite® 1958 produced the highest l rpm viscosity in the formulations where Tertiary-Butyl Acetate (TBAc) was substituted for Mineral Spirits. In the Stormer viscosity range (see Table 3), the viscosities are virtually equal. And the Leneta sag results, NY leveling, 60_ Gloss, and Heat Aging results are all very similar. Thus in this medium oil alkyd formulation containing TBAc, Claytone® 40, Garamite® 1958 and Garamite® LS all seem to function reasonably.

Table 3: Medium Oil Alkyd Results

Sample ID	Stormer (ku)	Leneta Sag	NY Leveling	60 Degree Gloss	Heat-Aging (60°C) Syneresis	
					1 week	2 weeks
KTO144093A	91	8	5	86.8	2.30	2.38
KTO144093B	72	10	4	91.0	3.37	3.49
KTO144093C	73	10	4	91.0	2.30	3.61
KTO144093D	70	10	5	92.7	1.16	2.38
KTO144093E	60	4	8	90.2	48.81	53.57

B. 100% Acrylic Formulations

The 100% acrylic portion of this evaluation uses two simple formulations. In the first formula, Paraloid B66 solid resin is first cut 50 / 50 with xylene. Once the cut is made, the thixotrope is incorporated at 0.3%. When the above pregel has dispersed for 10 minutes, the pigment (titanium dioxide) is then ground in for an additional 10 minutes at 4000rpm. The grind phase is then let down with xylene, toluene and methyl isoamyl ketone(MIAK). The MIAK is used as the tail solvent in this formulation to prevent the coating from drying too fast and causing film formation problems.

The second Acrylic formulation of this evaluation replaces the xylene with TBAc. This substitution reduces the VOC's of this coating from 5.61 lbs/gallon to 3.86 lbs/gallon. The MIAK remains as the tail solvent.

The results of the Acrylic formulations are included in Table 4 and Table 5.

Table 4: Brookfield Viscosities of 100% Acrylic Formulations

			Brookfield RVT (cps)		
Sample ID	Thixotrope	Solvent	1 rpm	10 rpm	100 rpm
KTO144099A	Control / No Thix	Xyl, Tol, MIAK	100	30	53
KTO144099B	Control / No Thix	TBAc, Tol, MIAK	100	30	74
KTO144099C	Garamite 1958	Xyl, Tol, MIAK	400	110	70
KTO144099D	Garamite 1958	TBAc, Tol, MIAK	1000	250	123
KTO144099E	Aerosil 200	Xyl, Tol, MIAK	1200	270	97
KTO144099F	Aerosil 200	TBAc, Tol, MIAK	1900	430	146
KTO144099G	Claytone APA	Xyl, Tol, MIAK	800	250	93
KTO144099H	Claytone APA	TBAc, Tol, MIAK	500	210	122

Aerosil 200 produced the highest low rpm viscosities in both of the acrylic formulations. However, as indicated in Table 5, Garamite® 1958 produces a higher sag result even with a lower viscosity. This is comparable to the performance of Garamite® 1958 in unsaturated polyester resin formulations, which has allowed VOC-compliant formulations to be developed. In the formulations with xylene, the system flocked in both the formulations with Aerosil 200 or Claytone® APA, resulting in very poor sag and syneresis results. Thus high viscosity does not necessarily equate to good rheological performance. The 60_ gloss results were negatively impacted by the inclusion of any thixotrope to the system, and the impact was greater on gloss in the system with TBAc. With a combination of low viscosity, good

sag, little syneresis, no settling, and the least negative impact on gloss, Garamite® 1958 produces excellent properties in the Acrylic system containing TBAc.

Table 5: 100% Acrylic Results

Thixotrope	Stormer (ku)	Leneta Sag	60° Gloss	Syneresis		Heat-Aging (60°C) Settling	
				4 days	2 weeks	4 days	2 weeks
Control / No Thix	<53	0 - runs	82.6	<1%	<1%	hard	hard
Control / No Thix	<53	0 - runs	82.9	<1%	5.19%	hard	hard
Garamite 1958	<53	4 - runs	80.4	7.32%	7.50%	none	none
Garamite 1958	54	6 - no runs	67.4	4.71%	5.00%	none	none
Aerosil 200	<53	0 - runs (flocked)	62.2	60.49%	65.43%	soft	soft
Aerosil 200	54	5 - no runs	41.9	32.14%	58.33%	soft	soft
Claytone APA	<53	4 - runs (flocked)	68.9	61.90%	73.68%	soft	soft
Claytone APA	53	5 - runs	49.6	65.00%	68.75%	soft	soft

Conclusions:

Solvent-based coatings, when properly formulated with exempt solvents, can be as environmentally friendly as their waterborne versions. The combination of TBAc and Garamite® 1958 gives formulators additional tools to achieve VOC and HAP compliance without sacrificing performance. When alternative solvents are desirable, Garamite® 1958 is compatible with many polar and aromatic systems; see Table 6. With the following features and benefits, Garamite® 1958 is proving to be valuable in many new systems:

FEATURES	BENEFITS
Efficient in high solids and 100% solids formulations.	VOC-compliant formulations.
Ease of dispersion.	Less mixing time, increased throughputs, decreased manufacturing costs.
High bulk density and low dust.	Ease of handling and less warehouse space.
Good syneresis control and viscosity stability.	Product consistency and customer satisfaction.
Lower system viscosities, good sag resistance and shear thinning.	Better application and surface appearance.

Each of the solvents listed below were used on an equal volume basis. To each solvent amount, 40 grams of Aroplaz 663-X-50 resin was added; this is a†phenolic alkyd resin cut in 50% xylene. To this mixture, 14 grams of Garamite® 1958 was added. This mixture was high speed dispersed for 10 minutes at 2000 rpm on a dispermat using a 40 mm blade. The sample was covered and allowed to set at ambient temperature for 7 days. The Brookfield viscosities were then measured.

Table 6: Performance of Garamite® 1958 in Various Solvents

Solvent	Brookfield Viscosity		
	1 RPM	10 RPM	100 RPM
Toluene	29600	8800	1656
n Butyl Propionate	23200	13880	2200
Xylene	8200	3860	997
Aromatic 100	300	1500	518
Aromatic 150	1200	1950	729
Oxsol 100	10800	3260	546
N-Butanol	6400	4320	588
N-Propyl Alcohol	2200	1600	238
Diacetone Alcohol	43600	6400	776
Acetone	4400	1390	155
Methyl Ethyl Ketone	30800	3620	428
Methyl Isobutyl Ketone	29600	12120	1380
Dissobutyl Ketone	47000	16200	2550
Methyl Iso Amyl Ketone	27200	11600	1608
Methyl n-Amyl Ketone	44000	12160	1820
Cyclohexanone	39200	5560	788
Exxate 800	42000	12520	1724
Butyl Cellosolve	48400	6520	836
Butyl Acetate	30000	15000	2090
PM Acetate	3400	2240	282
Isobutyl Acetate	80000	11200	2520
Methylpyrrolidone	9800	1440	254
Downal PM	8000	2060	246
Dow Propyl Propasol	137000	23600	4050

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