

Bio glycerol in air drying and stoving alkyds

General

This Technical Information leaflet presents low cost air drying/ stoving alkyds intended for decorative and protective coatings. Results relate to drying properties, hardness, resistance and esterification time wholly comparable to those of alkyds based on standard polyols.

Bio glycerol is a by-product of bio refinery Rapeseed oil, Sweden.

Comments

In air drying as well as stoving, the performance of the bio glycerol-based alkyds is comparable to standard glycerol in terms of hardness, chemical and mechanical properties

Though the initial colour of bio glycerol is much higher, the final color of the alkyds is slightly higher than standard glycerol based alkyds. However white paint based on both is found to be comparable
Bio glycerol should be used on a purity basis for better processing parameters.

Results

- Perstorp's bio glycerol is well suited for producing alkyds
- Coatings properties similar to standard glycerol grade
- Slightly higher colour on alkyds, but no visible difference in paints
- Should be used on a purity basis

Bio glycerol in medium oil length soybean oil-based air drying alkyd resin formulation

Resin formulations (Oil length 49.4 %)

Materials	Weight %		
	A	B	C
Bio Glycerol *	--	17.20	17.20/(17.554)
Glycerol- Std	17.20	--	--
Phthalic anhydride *	36.65	36.65	36.65
Soybean oil	49.40	49.40	49.40
Benzoic acid	1.00	1.00	1.00
Total charge	104.25	104.25	104.25
Reaction water	4.25	4.25	4.25
Yield	100.00	100.00	100.00

* Perstorp AB, Perstorp Specialty Chemicals

† 2 % Excess Bio glycerol (on the basis of weight of Polyol in the formulation)

Formulation data	A	B	C
Theoretical acid number	11.0	11.0	11.0
Oil length, %	49.4	49.4	49.4
Hydroxyl-number, mg KOH/g	43.2	43.2	43.2
Hydroxyl excess, %	8.54	8.54	8.54
Molecular weight, g/mole	6510	6510	6510
Alkyd constant	0.994	0.994	0.994
Acid Number GEL mg KOH/g	2.40	2.40	2.40
Functionality	2.013	2.013	2.013
Practical data			
Final acid Number, mg KOH/g	12.1	12.1	12.0
Esterification temp, °C	230	230	230
Esterification time, hrs	2 hr 50 min	3 hr 10 min	3 hr 10 min
Color on Gardner @ 50%NVM *	9.7	11.0	10.3
Viscosity, mPas @ 23°C, 50% NVM*	4499	5015	3095

* in white spirit

Synthesis procedure

Processing under nitrogen purge and xylene as azeotropic solvent

- Charge the oil
- Heat the oil to 160 °C
- Charge Polyols and alcoholysis catalyst (Lithium hydroxide 0.04% of oil quantity)
- Raise the temperature to 240°C. Hold until the alcoholysis is completed.
- Cool to 160°C and charge remaining raw materials and xylene
- Again raise the temperature for 1.5 hr to 230°C. Hold until desired acid number is obtained.
- Cool and dilute in white spirit*

*White spirit from HPCL, India

Lacquer formulation

Materials	Weight %
Alkyd 50% in white spirit	95.98
Cobalt Octoate (6%)	0.40
Calcium Octoate (6%)	1.60
Zirconium Octoate (6%)	2.00
Total charge	100.00

Performance Properties of Air Drying Lacquer (Oil length 50%)

Air drying on glass panels at 23°C & Relative Humidity 55 – 65%

König pendulum hardness			
Time after application	A	B	C
1 day	32	31	32
3 days	45	43	46
7 days	46	43	46
14 days	57	62	57
21 days	60	62	62
28 days	60	62	62
35 days	60	63	62
After 35 days Water 2 hrs, König sec's	54	56	56
After 21 days Water 2 hrs, Appearance ^{*1}	0	0	0
After 21 days Ethanol 2 hrs, Appearance ¹	1	3	2
Film thickness (dry) ± 2 mm	30	30	30
Thumb dry test	3 hr 45 min	5 hr	4 hr

*1 Density/size acc. To SS 184202 part 2 "Designation of degree of blistering" – Identical with ISO 4628/2-1982, 0=No blistering

Resin formulations Based on Soybean Oil and Coconut Oil (Oil length 40 and 42 %)

Materials	Weight %			
	A	B	C	D
Bio Glycerol *1	--	22.23	--	21.51
Glycerol-std	22.23	--	21.51	--
Benzoic acid	0.50	0.50	--	--
Phthalic anhydride *1	42.00	42.00	41.21	41.21
Soybean Oil	40.00	40.00	--	--
Coconut Oil	--	--	42.00	42.00
Total charge	104.23	104.23	104.72	104.72
Reaction water	4.23	4.23	4.72	4.72
Yield	100	100	100	100

*1 Perstorp AB, Perstorp Specialty Chemicals

Formulation data	A	B	C	D
Acid number	14	14	9	9
Oil length, %	40	40	42	42
Hydroxyl number, mg KOH/g	100	100	90	90
Hydroxyl excess, %	21.66	21.66	19.56	19.56
Molecular weight, g/mole	3501	3501	3118	3118
Alkyd constant	1.005	1.005	1.022	1.022
Acid number GEL, mg KOH/g KOH/g	-2.0	-2.0	-9.0	-9.0
Functionality	1.99	1.99	1.957	1.957
Practical Data				
Final acid Number, mg KOH/g	12.1	12.1	12.0	11.5
Esterification temp, °C	230	230	230	230
Esterification time, hrs	2 hr 50 min	3 hr 10 min	3 hr 10 min	2 hr 50 min
Color on Gardner, 50%NVM	6.9	7.1	6.1	6.5
Viscosity, mPas 23°C, 50% NVM in xylene	4499	5015	3095	2759

Synthesis procedure

Processing under nitrogen purge and xylene as azeotropic solvent

- Charge the oil
- Heat the oil to 160 °C
- Charge Polyols and alcoholysis catalyst (Lithium hydroxide 0.04% of oil quantity)
- Raise the temperature to 240°C. Hold until the alcoholysis is completed.
- Cool to 160°C and charge remaining raw materials and xylene
- Again raise the temperature for 1.5 hr to 230°C. Hold until desired acid number is obtained.
- Cool and dilute in xylene *

Lacquer formulation

Materials	Weight %
Alkyd 60% in mix Xylene	60.00
Butylated MF resin (60%)	25.00
Mix Xylene	15.00
Total charge	100.00

Lacquer properties

Curing on glass panel and steel panel 30min at 130°C

König hardness	A	B	C	D
	76	77	46	52

Paint Formulation

Ingredients	PBW
Mill Base	
Alkyd resin (@60% NVM)	18.00
TiO2 Rutile	26.70
Mix-Xylene	5.00
DisperBYK 163	0.80
Total	50.50
Stablization	
Alkyd resin (@60% NVM)	4.00
Mix-Xylene	1.40
Total	55.70
Letdown & Thinning	
Alkyd resin (@60% NVM)	16.55
n-Butanol	7.50
Butylated MF Resin*	16.52
Dowanol PMAC	3.50
BYK-066N	0.03
Grand Total	100.00

* Blumer MF 1310, Bluebell Polymers Pvt. Ltd., India

Paint Preparation

Step –I Initial Grind

Disperse TiO2 in Alkyd resin containing dispersing agent under high speed mixer at 1200 rpm. Stir it for 10 minutes and add zirconium beads (grinding media) and stir it for 15-20 minutes at 1800 rpm. Check finish of grind on Hegman gauge. Finish should be a minimum of 7 on the gauge.

Step –II Stabilization

Add mix of alkyd resin and solvent slowly to stabilize the dispersed mill base at 1200 rpm. Stir for the next 5-10 minutes.

Step –III let down & thinning

Add remaining quantity of alkyd resin, Butylated MF resin and additives. Stir it for 5 - 10 minutes. Filter it through 40 Micron nylon mesh

Stoving white paint data	A	B	C	D
Alkyd : Melamine (solids)	70/30	70/30	70/30	70/30
PVC	10.77	10.77	10.77	10.77
Non volatile matter %	60.24	60.24	60.24	60.24
Viscosity 23°C in ford cup B4 (sec)	54	56	37	38
Grinding Speed Time-on High speed Stirrer	1800 rpm 30 min	1800 rpm 30 min	1800 rpm 30 min	1800 rpm 30 min

Paint Properties

Curing on glass panel and steel panel 30min at 130°C

Properties	A	B	C	D
König hardness, sec's @ 23°C & 55 - 65% RH	56	52	42	46
Methyl ethyl ketone double rub	32	35	10	14
Adhesion (cross-cut)	Passes	Passes	Passes	Passes
Impact reverse, inch-lbs (passes up to)	20	15	30	25
Pencil hardness	H-2H	H-2H	HB-H	HB-H
Flexibility on 1/4" Conical mandrel	Passes	Passes	Passes	Passes

Every endeavour has been made to ensure that the information given herein is true and reliable but it is given only for the guidance of our customers without any warranty and with reservation of all patent rights. Users are advised to confirm the suitability of our recommendations by their own tests.