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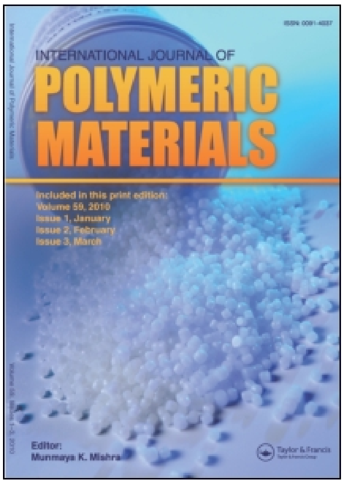

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R. P. Patel[a]; Pulin Patel[b]; D. A. Raval[b]
[a] Department of Industrial Chemistry, V.P. and R.P.T.P. Science College, Vallabh Vidyanagar [b] Department of Chemical Sciences, N. V. Patel College of Pure and Applied Sciences, Vallabh Vidyanagar

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Alkyd Resins from Acrylated Prepolymerised Rubberseed Oil

R. P. PATEL[a], PULIN PATEL[b] and D. A. RAVAL[b,*]

[a]*Department of Industrial Chemistry, V.P. and R.P.T.P. Science College, Vallabh Vidyanagar - 388120;* [b]*Department of Chemical Sciences, N. V. Patel College of Pure and Applied Sciences, Vallabh Vidyanagar - 388120*

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Rubberseed oil (RSO) was polymerised at different temperatures *viz.* 60, 90 and 230°C using various percentages of benzoyl peroxide (BPO) and Azobisisobutyronitrile (AIBN) as free radical catalysts. This polymerised RSO was then subjected to acrylation by using MMA as an acrylic monomer and BPO as free radial initiator. The viscosities of the acrylated oil samples were examined.

Alkyd resins were prepared from as is and acrylated RSO samples. The physical properties and air dried and baked film properties of these alkyd resins were studied and compared.

Keywords: Alkyd resins; Rubberseed oil; Acrylates; Physical properties

1. INTRODUCTION

Among the oldest binders for coating are oils which can react with oxygen to form solid films. They have been used since prehistoric times and in the nineteenth and early twentieth centuries, binders of most paints were drying oils [1]. They are the important raw materials for other binders such as alkyd resins, epoxy esters and uralkyds. Oils govern many physical and chemical properties of the resultant resin (binder). The properties of the resin depend on the initial properties of the oil. Several types of modifications of oils have been practised to

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*Corresponding author.

improve the initial properties [2]. In our earlier paper [3], we reported the modification of oil by subjecting it to catalytic and thermocatalytic polymerisation by utilizing BPO and AIBN as free radical initiators. In the present study, an attempt has been made to further improve the properties of thermocatalytically polymerised oil by acrylation.

Various acrylated polymerised oil samples have been used in the synthesis of medium oil alkyd resin by following monoglyceride process using calcium octoate as a catalyst. The various physical properties of these alkyd resins have been studied and compared.

2. MATERIALS AND METHODS

Raw rubberseed oil was procured from Kottayam, Kerala state, India and was purified prior to its use. Its characteristics are shown in Table I. The other chemicals used *viz.* glycerol, phthalic anhydride, BPO, AIBN, litharge lead oxide (PbO), calcium octoate, xylene, methanol and phosphoric acid were of laboratory reagent grade.

Acrylic monomer MMA was procured from M/s Gujarat State Fertilizers and Chemicals Ltd. (Polymers unit) Petrofils, Baroda (India). Driers used were commercial samples.

The coating compositions were applied on 6″ × 2″ mild steel panels and tinned steel panels as specified in Indian Standard Specifications [4] to cast films having dry film thickness of approximately 30 microns.

TABLE I Characteristics of RSO

Sr. no.	*Properties*	
1	Acid value (mg. KOH/gm)	9.00
2	Saponification value (mg. KOH/gm)	193.00
3	Iodine value	138.00
4	Hydroxyl value	20.00
5	Specific gravity (15°C)	0.925
6	Refractive Index	1.4670
7	Colour [$y = 5R$ (Lovibond)]	11.00

The mechanical properties of the films such as flexibility, impact resistance, scratch hardness and adhesion have been determined as per Indian Standard Specifications [4]. The chemical resistance properties [5] *viz.* acid and alkali resistance of the film were studied by immersion of the coated panels in 2% by wt. of solution of HCl and NaOH respectively.

3. EXPERIMENTAL

Rubberseed oil was refined by the reported method [6].

3.1. Polymerisation of Rubberseed Oil

Refined rubberseed oil (RSO) was heated at different temperatures *viz.* 60, 90 and 230°C using (w/w) of the oil. 0.5, 1.0, 1.5 and 2.0% of AIBN and BPO separately and respectively as catalyst and without using the catalysts. The temperatures were controlled within ± 2°C. The polymerised oil was taken out after 1, 2, 3 and 4 hrs. cooled and was fractionated by shaking one part of the polymerised oil and two parts of *n*-hexane at room temperature for 15 minutes and allowing it to settle for 30 minutes. The two layers were separated. The hexane soluble layer which contains the polymerised oil was decanted off completely from the extract.

Viscosity and refractive index of the hexane extracted fractions were determined by Gardner Bubble Viscometer and Abbe's Refractometer respectively. The other properties were determined as per AOCS method [7].

3.2. Acrylation of Polymerised Rubberseed Oil

The acrylation of the polymerised oil was carried out by the reported method [8]. MMA was added in three different percentages *viz.* 5, 15, 25 w/v% of the oil.

After acrylation the samples were tested for viscosity, colour *etc.* Their values are reported in Table II.

TABLE II Characteristics of acrylated RSO(P)

Resin code	*% age AIBN/BPO*	*% MMA*	*Viscosity*	*Time of acrylation Hrs.*	*Temp.°C*
RSO(P)-I, V	0.5	5	D		
		10	D-E		
		15	E	4	90
		20	E-F		
		25	GELLED		
RSO(P)-II, VI	1.0	5	D-E	4	90
		10	D-E		
		15	E-F		
		20	F		
		25	GELLED		
RSO(P)-III, VII	1.5	5	D-E	4	90
		10	E		
		15	E-F		
		20	F		
		25	GELLED		
RSO(P)-IV, VIII	2.0	5	D-E	4	90
		10	E		
		15	F		
		20	GELLED		
		25	GELLED		

4. PREPARATION OF ALKYDS

The alkyds from modified RSO were prepared by the conventional solvent process [9]. Modified RSO (50 gms) was heated to 100°C in a multineck reaction flask, provided with a mechanical stirrer, a Dean and Stark trap and thermometer. Glycerol (17 gm), litharge (0.04% by wt. of the total charge) and calcium octoate (0.4% by wt. of total charge) were added together. Temperature was raised to 240°C and maintained at this value until a sample of the product appeared clear when mixed with methanol (1 : 3 by volume) at room temperature, indicating the formation of the monoglyceride (MG).

After cooling the reaction mixture to 100°C, phthalic anhydride (PA, 33 gm) was added along with xylene (8% by wt. of the total charge). Temperature was then raised to 160°C, maintained for an hour and again raised to 220°C during which the solvent was stripped off. This temperature was maintained until desired acid value was reached (< 15). The cooled resin was thinned with xylene to 60%

nonvolatile content. The characteristics of the resulting resins are shown in Table III.

4.1. Film Preparation

Air dried and baked (150°C for 15 min) film properties of various alkyds were studied. Driers were added to the samples (Pb – 0.5%,

TABLE III Characteristics of alkyd resins

Resin code	*% AIBN*	*% MMA*	*Colour*	*Viscosity*	*Acid value mg KOH/g*
RSO(P)-I	0.5	5	15	E	12.0
		10	14–15	E–F	12.5
		15	14	F	13.7
		20	13–14	G	14.8
RSO(P)-II	1.0	5	15–16	F	14
		10	15	F–G	14.5
		15	15	G	14.8
		20	14–15	G–H	15.0
RSO(P)-III	1.5	5	16	F–G	14.8
		10	15–16	G	15.0
		15	15	G–H	15.1
		20	14–15	I	15.6
RSO(P)-IV	2.0	5	16	F–G	15.0
		10	15–16	G–H	15.5
		15	15	H	15.8
		20	–	–	–
Resin code	*% BPO*	*% MMA*	*Colour*	*Viscosity*	*Acid value mg KOH/g*
RSO(P)-V	0.5	5	15	E	12.5
		10	14–15	E–F	12.8
		15	14	F	13.6
		20	14	G	15.0
RSO(P)-VI	1.0	5	16	F	14.6
		10	15–16	F–G	14.8
		15	15	G	14.8
		20	14–15	G–H	15.0
RSO(P)-VII	1.5	5	16–17	F–G	14.7
		10	16	G	15.0
		15	15–16	G–H	15.5
		20	15	I	16.0
RSO(P)-VIII	2.0	5	16	F–G	15.0
		10	15–16	G–H	15.6
		15	15	H	16.0
		20	–	–	–

Co – 0.05% and Mn – 0.05%) and viscosity of the composition was adjusted suitably by adding xylene. The coating compositions were applied on 6″ × 2″ mild steel panels and tin panels.

5. RESULTS AND DISCUSSION

5.1. Acrylated Oil and Alkyds

The characteristics of various acrylated oils and alkyd resins are listed in Tables II and III respectively.

In general, for acrylated oil, for both the catalysts used for polymerisation of the oil, the viscosity of the oil increases with increase in degree of acrylation. This can be attributed to the inclusion of acrylic monomer in the oil.

The colour of the alkyd was also found to be improved as compared to the alkyd from polymerised oil. This can be attributed to the inclusion of acrylic monomer in the oil moiety.

5.2. Drying Time

The results of air dried and baked film properties are listed in Tables IVA and IVB. In general, drying time of the films from acrylated RSO is less than that of alkyd from nonacrylated polymerised RSO and unpolymerised RSO. The drying time of the alkyd from acrylated RSO (P) decreases with the increase in percentage of acrylic monomer. This should be due to increase in acrylic monomer into the oil moiety. Acrylic monomers are known to increase the molecular weight [11]. Therefore, with increase in molecular weight of the oil, fewer crosslinks are required to form a coherent film [12] or to reach the dry stage [13]. Drying time for alkyds prepared from RSO (P) and RSO is high as compared to alkyd prepared from acrylated RSO (P). This might be due to difference in molecular weight, which is reflected in the viscosities.

5.3. Mechanical Properties

In general, for both air dried and baked films, the scratch and impact resistance of the alkyd prepared from acrylated RSO (P) are better

TABLE IVA Air dried film properties of alkyd resin

Resin code	*AIBN*	*Drying time* *Surface dry*	*Drying time* *Hard dry*	*Scratch hardness (passes) Kg.*	*Impact resistance lbs inch*	*NaOH* (2%) *(passes) hr.min*
RSO(P)-I	1	1.25	7.55	1.0	250	0.55
	2	1.15	7.50	1.1	275	1.00
	3	1.05	7.35	1.3	300	1.05
	4	1.00	7.25	1.1	275	1.10
RSO(P)-II	1	1.15	7.45	1.1	275	1.00
	2	1.05	7.35	1.3	300	1.00
	3	0.55	7.25	1.7	300	1.10
	4	0.50	7.20	1.4	275	1.15
RSO(P)-III	1	1.05	7.35	1.4	300	1.00
	2	0.55	7.30	1.8	300	1.05
	3	0.50	7.20	2.0	300	1.10
	4	0.45	7.05	1.7	275	1.15
RSO(P)-IV	1	0.55	7.25	1.5	300	1.05
	2	0.45	7.15	1.8	300	1.10
	3	0.35	7.10	2.2	275	1.20
	4	–			–	–

Resin code	*BPO*	*Drying time* *Surface dry*	*Drying time* *Hard dry*	*Scratch hardness (passes) Kg.*	*Impact resistance lbs inch*	*NaOH* (2%) *(passes) hr.min*
RSO(P)-V	1	1.30	8.00	1.0	250	0.55
	2	1.25	7.50	1.1	275	1.00
	3	1.15	7.35	1.3	300	1.05
	4	1.05	7.25	1.1	275	1.10
RSO(P)-VI	1	1.20	7.50	1.1	275	1.00
	2	1.15	7.35	1.3	300	1.00
	3	1.05	7.25	1.7	300	1.00
	4	0.55	7.20	1.4	275	1.15
RSO(P)-VII	1	1.10	7.40	1.4	300	1.00
	2	1.00	7.30	1.8	300	1.05
	3	0.50	7.20	2.0	300	1.10
	4	0.45	7.05	1.7	275	1.15
RSO(P)-VIII	1	1.0	7.30	1.5	300	1.05
	2	0.50	7.25	1.8	300	1.10
	3	0.40	7.15	2.2	275	1.20
	4	–	–		–	–

than those from the RSO and RSO(P). The scratch and impact resistance increase with increase in acrylic monomer loading in the oil. This might be due to increase in initial molecular weight of the oil which is reflected in the resulting alkyd. The improvement in

TABLE IVB Baked film properties of alkyd resins

Resin code	*AIBN*	*Scratch hardness*	*Impact resistance lbs inch*	*NaOH* (2%) (*passes*) *hr.min*
RSO(P)-I	1	1.2	275	1.05
	2	1.4	300	1.10
	3	1.7	300	1.15
	4	1.5	275	1.25
RSO(P)-II	1	1.4	275	1.10
	2	1.8	300	1.15
	3	2.1	300	1.25
	4	1.9	275	1.30
RSO(P)-III	1	1.6	300	1.15
	2	2.0	300	1.25
	3	2.4	300	1.30
	4	2.0	250	1.35
RSO(P)-IV	1	2.0	275	1.30
	2	2.5	300	1.40
	3	2.7	300	1.50
	4	–	–	–

Resin code	*BPO*	*Scratch hardness*	*Impact resistance lbs inch*	*NaOH* (2%) (*passes*) *hr.min*
RSO(P)-V	1	1.2	275	1.05
	2	1.4	300	1.10
	3	1.7	300	1.15
	4	1.5	275	1.25
RSO(P)-VI	1	1.4	275	1.10
	2	1.8	300	1.15
	3	2.1	300	1.25
	4	1.9	275	1.30
RSO(P)-VII	1	1.6	300	1.15
	2	2.0	300	1.25
	3	2.4	300	1.30
	4	2.0	250	1.35
RSO(P)-VIII	1	2.0	275	1.30
	2	2.5	300	1.40
	3	2.7	300	1.50
	4	–	–	–
RSO(P)	–	–	–	–

mechanical properties of the modified alkyd can also be attributed to the inclusion of acrylic polymer in the alkyd moiety [11].

The examination of results also reveals that the impact resistance and scratch hardness increases up to 15% loading of MMA. However above 15%, the impact resistance and scratch hardness decrease which can be attributed to increase in glass transition temperature of the resin. Since MMA increases the Tg, the film becomes more brittle. Similar results were obtained for all the samples which were prepared from the polymerised oil using AIBN and BPO as free radical initiators for polymerisation of the oil.

5.4. Chemical Resistance

The alkali resistance of the alkyds prepared from acrylated RSO (P) is better than that of the alkyds prepared from RSO (P) and RSO. The alkali resistance for air dried and baked films increases with increase in acrylic monomer loading. Acrylic monomers have good chemical resistance properties which are reflected in the chemical resistance tests *viz.* acid, water and xylene show that alkyds from acrylated RSO (P) have better resistance towards these chemicals than the one from RSO (P) and RSO.

The characteristics of RSO (P), characteristics of alkyd prepared from RSO (P) and their film properties are shown in Tables VA, VB, VI, VIIA and VIIB respectively. These results had been the part of our earlier paper [3].

TABLE VA Characteristics of polymerised rubberseed oil

Catalyst	*Concentration of catalyst (%)*	*Time of polymerisation (hour)*	*Iodine value*	*Acid value (mg of KOH/gm)*	*Viscosity (Gardner)*	*R.I at 25°C*	*Temperature°C*
BPO	0.5	1.0	122	11.8	D	1.4978	230
		2.0	119	14.1	D–E	1.5148	
		3.0	115	18.2	E	1.5207	
		4.0	110	20.2	F	1.5248	
	1.0	1.0	116	14.2	D–E	1.5141	
		2.0	113	17.0	D–E	1.5197	
		3.0	109	20.1	E–F	1.5250	
		4.0	106	24.8	F	1.5298	
	1.5	1.0	112	15.0	D–E	1.5204	
		2.0	108	18.3	D–E	1.5255	
		3.0	105	21.8	F	1.5301	
		4.0	103	27.7	F–G	1.5344	
	2.0	1.0	108	16.2	E	1.5244	
		2.0	106	19.8	E–F	1.5300	
		3.0	102	23.4	E–F	1.5354	
		4.0	99	29.2	G	1.5402	

TABLE VA (Continued)

Catalyst	Concentration of catalyst (%)	Time of polymerisation (hour)	Iodine value	Acid value (mg of KOH/gm)	Viscosity (Gardner)	R.I at 25°C	Temperature°C
AIBN	0.5	1.0	120	10.3	D	1.4975	230
		2.0	118	13.2	D–E	1.5145	
		3.0	115	17.3	E	1.5201	
		4.0	110	19.4	F	1.5244	
	1.0	1.0	115	13.3	D–E	1.5139	
		2.0	112	16.5	D–E	1.5197	
		3.0	109	19.3	E–F	1.5247	
		4.0	106	14.2	F	1.5296	
	1.5	1.0	110	14.5	D–E	1.5202	
		2.0	107	17.5	D–E	1.5255	
		3.0	105	20.3	F	1.5301	
		4.0	102	26.3	F–G	1.5342	
	2.0	1.0	108	15.3	E	1.5222	
		2.0	105	18.5	E–F	15299	
		3.0	101	22.3	E–F	1.5351	
		4.0	98	28.4	G	1.5400	
–	–	1.0	137	9.00	B–C	1.4670	230
		2.0	137	9.00	C	1.4700	
		3.0	136	9.50	C–D	1.4700	
		4.0	134	10.00	C–D	1.4750	

TABLE VB Characteristics of polymerised rubberseed oil

Catalyst	Concentration of catalyst (%)	Time of polymerisation (hour)	Iodine value	Acid value (mg of KOH/gm)	Viscosity (Gardner)	R.I at 25°C	Temperature°C
BPO	0.5	1.0	135	9.08	B	1.4673	90
		2.0	133	9.18	B–C	1.4690	90
		3.0	130	10.00	C	1.4800	90
		4.0	129	10.08	C–D	1.4890	90
	1.0	1.0	133	9.25	B–C	1.4705	90
		2.0	130	9.50	C	1.4785	90
		3.0	127	10.18	C–D	1.4810	90
		4.0	122	10.25	D	1.4900	90
	1.5	1.0	130	9.38	B–C	1.4780	90
		2.0	125	9.60	C	1.4809	90
		3.0	123	10.20	C–D	1.4910	90
		4.0	119	10.30	D–E	1.5011	90
	2.0	1.0	127	9.50	C	1.4880	90
		2.0	124	9.75	C–D	1.4910	90
		3.0	120	10.30	D	1.5018	90
		4.0	118	10.48	D–E	1.5105	90
	0.5	1.0	132	9.06	B	1.4670	60
		2.0	130	9.15	B–C	1.4687	60
		3.0	127	9.78	C	1.4795	60
		4.0	124	10.00	C–D	1.4882	60
	1.0	1.0	130	9.15	B–C	1.4700	60
		2.0	127	9.38	C	1.4779	60
		3.0	123	9.88	C–D	1.4806	60
		4.0	120	10.06	D	1.4896	60

TABLE VB (Continued)

Catalyst	*Concentration of catalyst (%)*	*Time of polymerisation (hour)*	*Iodine value*	*Acid value (mg of KOH/gm)*	*Viscosity (Gardner)*	*R.I at 25°C*	*Temperature°C*
AIBN	1.5	1.0	129	9.25	B-C	1.4776	60
		2.0	126	9.50	C	1.4803	60
		3.0	123	9.98	C-D	1.4906	60
		4.0	118	10.18	D-E	1.5006	60
	2.0	1.0	126	9.40	C	1.4876	60
		2.0	123	9.70	C-D	1.4900	60
		3.0	120	10.00	D	1.5006	60
		4.0	117	10.35	D-E	1.5100	60
–	–	1.0	138	9.00	B	1.4670	60/90
		2.0	138	9.00	B	1.4670	60/90
		3.0	138	9.00	B	1.4673	60/90
		4.0	138	9.00	B-C	1.4680	60/90

TABLE VI Characteristic of alkyd resin*

Resin code	*Colour (Gardner)*	*Acid value (mg. KOH/gm)*	*Catalyst*	*Catalyst percentage*	*Time of polymerisation*
RSO(P)-I	15	11.2	RSO	0.5	4 hr
RSO(P)-II	15-16	13.6	RSO	1.0	4 hr
RSO(P)-III	16	14.9	RSO	1.5	4 hr
RSO(P)-IV	16	15.0	RSO	2.0	4 hr
RSO(P)-V	15	10.0	AIBN	0.5	4 hr
RSO(P)-VI	15-16	12.5	AIBN	1.0	4 hr
RSO(P)-VII	16	12.3	AIBN	1.5	4 hr
RSO(P)-VIII	16	15	AIBN	2.0	4 hr
RSO	14	8.1	–	–	–
RSO(pwc)	15	10.08	–	–	4 hr

RSO(P) = Polymerised rubber seed oil.
RSO = unpolymerised rubber seed oil.
RSO(pwc) = RSO polymerised without catalyst.

TABLE VIIA Air dried film properties of alkyd resins*

	Drying time				
Resin code	*Dry to touch hr.min*	*Tack free hr.min*	*Scratch hardness kg (passes)*	*Impact resistance lbs inch*	*Chemical resistance (unaffected) NaOH (2%) (passes) hr.min*
RSO(P)-I	1.40	8.30	0.8	250	0.45
RSO(P)-II	1.25	7.30	0.9	275	0.50
RSO(P)-III	1.10	6.45	1.0	300	1.00
RSO(P)-IV	1.00	6.00	1.1	300	1.10
RSO(P)-V	1.35	8.15	0.7	250	0.45
RSO(P)-VI	1.20	7.20	0.8	275	0.50
RSO(P)-VII	1.05	6.30	0.85	300	0.55
RSO(P)-VIII	0.50	5.50	0.90	300	1.10
RSO	2.00	NOT	0.7	225	0.35
RSO(pwc)	1.50	NOT	0.7	225	0.40

* The test panels were cured at ambient temperature.
The test panels pass the test of HCl (2%), water and xylene for 200 hr. immersion.

TABLE VIIB Baked film properties of alkyd resin*

Resin code	*Scratch hardness kg (passes)*	*Impact resistance lbs inch*	*Chemical resistance (unaffected) NaOH (%) (passes) hr.min*
RSO (P)-I	1.00	250	1.00
RSO (P)-II	1.10	275	1.10
RSO (P)-III	1.20	300	1.25
RSO (P)-IV	1.30	300	1.45
RSO (P)-V	0.50	250	1.00
RSO (P)-VI	1.00	275	1.10
RSO (P)-VII	1.10	300	1.25
RSO (P)-VIII	1.20	300	1.45
RSO	0.90	250	0.50
RSO (pwc)	0.90	250	0.50

* Baked at 150°C for 15 min.
The test panels pass the test of HCl (2%), water and xylene for 200 hr. immersion.

6. CONCLUSION

The acrylation of the polymerised RSO can be carried out successfully and the overall properties of the oil and alkyd can be improved.

From the results, it is also concluded that there is an optimum modification of the oil by MMA, beyond which the mechanical properties of the alkyd get adversely affected.

The modified alkyds can be used as binders for industrial maintenance coating.

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