# Full Length Research Paper

# Effect of modification with nitrocellulose and phenol formaldehyde resins on some performance characteristics of rubber seed oil alkyds

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Accepted 7 March, 2006

Three alkyd samples each of 50% oil length were prepared with phthalic anhydride, glycerol and crude rubber seed oil (I), refined rubber seed oil (II) and methyl esters of rubber seed oil (III) employing the alcoholysis method. All the alkyds were processed to acid value below 10 mg KOH/g. The alkyd samples were blended with nitrocellulose and phenol formaldehyde resins in different proportions. The performance of the blends in terms of drying schedule, chemical resistance and pencil hardness was determined. The blends were observed to show improved properties to a substitution level of 40% rubber seed oil alkyd in the blend. The resistance of the blends to different service media was found to be better than the rubber seed oil based alkyd resins.

Key words: Alkyd resin, Rubber seed oil, Methyl ester, Phenol formaldehyde, Nitrocellulose

### INTRODUCTION

One of the unique properties of alkyd resins which make them versatile binders in surfaces coatings is their compatibility with other film formers like nitrocellulose resins (Waldie, 1983). Blending of alkyds with other resins vields new binder which combines advantageous qualities of the alkyd and the blended resins. The wide spectrum of properties of alkyd resins is broadened by modification with a variety of materials like nitrocellulose, phenolic resin, etc. This fact has vastly extended the use of alkyd resins to an extent that any new film-former developed is checked for compatibility with various types of alkyd resins (Maron et al., 2002; Aigbodion et al., 2001). Generally, the alkyd contributes to coating flexibility, adhesion, durability, and gloss; while the other resins e.g. phenolic resins, confers faster drying rate, improved film hardness and resistance to chemicals. Resins chemists take advantage of the compatibility property of alkyd to prepare new set of binders that meet

certain specialized end-use specifications (Zeno et al., 1999; Anthawale et al., 2000; Anthawale and Chamanker, 2000).

In this study, samples of rubber seed oil modified alkyd (50% oil length) were blended at various proportions with nitrocellulose and phenol formaldehyde resins. Nitrocellulose resins are fast drying but brittle; while phenolic resins are highly water and alkali resistance. Rubber seed oil-modified alkyd resins have been recognized to posses strong potential for use as binders in surface coatings (Aigbodion and Pilla, 2000; Ikhuoria et al., 2005; Ikhuoria and Aigbodion, 2005). This study was therefore undertaken to explore both the technical and economic benefits of blending rubber seed oil alkyd resins with commercial grade binders commonly used in the Nigeria surface coating industry.

## MATERIALS AND METHODS

Rubber (*Hevea brasiliensis*) seed oil was extracted by solvent methods from the dry seed employing n-hexane. Phthalic anhydride, glycerol, xylene and other chemicals used were of industrial grade.

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Ratio of PF: RSO alkyd	Drying schedule										
		Set-to-touch (m	in)	Dry through (min)							
	I	II .	III	I	11	111					
0:1	57	62	65	>16hrs	>16hrs	>16hrs					
1:4	46	40	56	840	720	>16hrs					
1:1	35	31	45	720	720	720					
3:2	29	25	32	360	360	720					
4:1	18	22	23	120	120	360					
1:0	15	15	15	120	120	120					

Table 3. Effect of blending with phehol formalhyde (PF) resin on the drying schedule of RSO alkyd samples I –III.

Table 1. Recipe for RSO alkyds

Ingredient (g)	Alkyd samples							
	ı	II	III					
Rubber seed oil								
Crude	151.13	-	-					
Refined	-	151.13	-					
Methyl ester	-	-	124.92					
Phthalic anhydride	91.28	91.28	43.13					
Glycerol	57.60	57.60	47.95					

Commercial grade nitrocellulose (NC) and phenol formaldehyde (PF) resins were obtained from Boston Atlantic Co. Ltd., Benin City.

**Table 2**. Effect of blending with nitrocellulose (NC) resin on the drying schedule of RSO alkyd samples I – III.

Ratio of NC:	Drying schedule									
RSO alkyd	Set-to	o touch	(min)	Dry through (min)						
	ı	II	III	1 11 111						
0:1	30	20	40	overnight						
1:4	6	6	10	60 60 60						
1:1	4	4	6	30	60					
3:2	2	2	6	30	60					
4:1	1	1	2	10 10 10						
1:0	1	1	<1	3 10 1						

### Refining of rubber seed oil

Rubber seed oil was alkali refined using the method adopted by (Cocks and Rede, 1966). In a typical batch, a known weight of the preheated oil (60°C) was treated with 10% (w/w) of 0.8 M sodium hydroxide by stirring for 5 min. After which it was allowed to stand or centrifuged to separate the aqueous layer. The oil phase was washed with water until neutral to give the refined oil.

### Preparation of methyl esters of rubber seed oil

The methyl ester was produced through the process of in-situ alcoholysis according to the method adapted from (Gökhan et al.,

1996). The rubber seed meal was macerated with methanol for 30 min and the slurry obtained was magnetically stirred under reflux for 3 h. At the end of the reaction, the mixture was vacuum-flitered, the residue washed with 200 ml methanol and air dried overnight at room temperature. The residue was re-extracted in a soxhlet apparatus with n-hexane to obtain the oil fraction remaining in the residue. To the filtrate obtained from the in-situ alcoholysis was added 100 ml of water and the solution re-extracted 3 times with n-hexane (3X 50 ml). The combined extracts were washed with water, dried over sodium sulphate and evaporated to give the esterified product.

### Preparation of the alkyd resin

Three alkyd samples each of 50% oil length were prepared with phthalic anhydride, glycerol, and crude rubber seed oil (I), refined rubber seed oil (II), and methyl esters of rubber seed oil (III) employing the alcoholysis method according to the procedure described elsewhere (Ikhuoria and Okieimen, 2005). The recipe used is shown in Table 1. Each alkyd was processed to acid value below 10 mg KOH/g.

The finished alkyds were blended separately with nitrocellulose and phenol formadehyde resins in the following ratios: RSO alkyd: commercial resin, 1:4, 1:1, 3:2 and 1:4. The alkyds and the blends were tested for their drying schedule, chemical resistance and film hardness using standard methods (ASTM 1640-83, 1991; ASTM D 1647-89, 1994; ASTM D 3363 74, 1979).

### **RESULTS AND DISCUSSION**

The effects of blending nitrocellulose (NC) and phenol formaldehyde (PF) resins on the drying schedule of the finished RSO based alkyd samples are given in Tables 2 and 3, respectively. It is observed in Table 2 that rate of drying become rapid with increase in the content of nitrocellulose resin in the blend. On the basis of the drying schedule, RSO based alkyd samples I – III could be blended with nitrocellulose resin in a ratio of 4:1. It can be inferred that RSO alkyds could be used to reduce production cost by substituting part of it with nitrocellulose resin in a formulation without compromising the rate of drying.

On the other hand, the results in Table 3 shows that PF resin is not as effective as nitrocellulose resins in improving the rate of drying of the RSO alkyd samples.

Ratio of NC: RSO alkyd	Chemical Resistance											
	Media											
	Distilled water			NaCl (5% Soln)			H <sub>2</sub> SO <sub>4</sub> (0.1 M)			KOH (0.1 M)		
0:1	2	2	1	1	1	1	1	2	2	5	3	3
1:4	2	2	1	1	1	1	2	2	2	3	2	2
1:1	2	2	1	1	1	1	2	2	2	2	2	2
3:2	1	1	1	1	1	1	1	1	1	2	2	2
4:1	1	1	1	1	1	1	1	1	1	1	1	1
1.0	-1	1	1	1 1	- 1	- 1	1	1	- 1	-1	1	- 1

Table 4. Effect of blending with nitrocellulose (NC) resin on the chemical resistance of RSO alkyds samples I – III.

Note: No effect = 1; whitening = 2; shrinkage of film=3; blistering of film=4 and Removal of film =5.

Table 5. Effect of blending with phenol formaldehyde (PF) resin on the chemical resistance of RSO alkyd samples I – III.

Ratio of PF: RSO alkyd	Chemical Resistance												
		Media											
	Distille	ed water	NaC	NaCl (5% Soln)			H <sub>2</sub> SO <sub>4</sub> (0.1 M)				KOH (0.1 M)		
0:1	2	2		1	1	1	2	2	2	5	3	3	
1:4	2	2	2	1	1	1	2	2	2	5	3	3	
1:1	2	2	2	1	1	1	2	2	2	2	2	2	
3:2	2	2	1	1	1	1	2	2	2	2	2	2	
4:1	1	1	1	1	1	1	1	1	1	2	2	2	
1:0	1	1	1	1	1	1	1	1	1	2	2	2	

Note: No effect = 1; whitening = 2; shrinkage of film=3; blistering of film=4; Removal of film =5.

However, PF resins enhance surface drying of RSO alkyd more than drying through.

The effects of blending nitrocellulose resin and phenol formaldehyde resin on the chemical resistance of RSO alkyd are shown in Tables 4 and 5, respectively. It can be seen in Table 4 that blending nitrocellulose resins with RSO alkyds improves their chemical resistance to different solvent media. All the blends and the individual resins are unaffected by 5% solution of NaCl. Blending with nitrocellulose resin enhances the low resistance of RSO alkyd samples to alkali solution. On economic consideration, it can be deduced that blending of nitrocellulose resin with RSO alkyd in a ratio 3:2 is economical.

Comparing the results in Table 4 with the results in Table 5, it is observed that PF resin is slightly less effective in enhancing the chemical resistance of alkyds compared to nitrocellulose resin. Good chemical resistance is obtained when PF resin and RSO alkyds are blended at a ratio of 4:1.

Pencil hardness tests of the films carried out show that blending of nitrocellulose and PF resins with RSO alkyd samples enhance hardness of the dry films. For the consideration of film hardness, nitrocellulose and RSO alkyd ratio 3:2; and PF resin and RSO alkyd ratio 4:1 are economical.

### Conclusion

Commercial grade nitrocellulose and phenol formaldehyde PF resins were blended in various proportions with RSO alkyd samples. The results obtained show that that rate of drying of RSO alkyds is greatly enhanced by blending with these commercial resins and blending with nitrocellulose is more effective than PF resins. Similarly, blending RSO alkyds with these commercial resins enhances chemical resistance and film hardness. Based on the results of this study, it is deduced that RSO alkyds can be blended with nitrocellulose and PF resins up to between 20 and 40% without deleterious effect on performance characteristics of the coatings. These findings will expand the field of applications of RSO alkyds

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