# Investigation of Drying and Chemical Properties of Unstyrenated and Styrenated Palm Kernel Oil Modified Alkyl Resins Using Some Empirical Models

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*Abstract:* **The investigation of drying properties of unstyrenated and styrenated palm kernel oil modified alkyd resins using some empirical equations was studied. Three different alkyd resins A, B and C were prepared with dehydrated palm kernel oil, phthalic anhydride, maleic anhydride and glycerol using lithium hydroxide as catalyst at stipulated ratios. The prepared alkyd resins were post- polymerized with styrene using benzoyl peroxide as an initiator under reflux at 120°C for 3 hours. The drying and chemical behaviour of the alkyd resins and styrenated alkyd resins were investigated using standard methods. Some empirical model equations (linear, exponential, logarithmic and quadratic) were tested for the dynamics of the physical drying and chemical resistance of both styrenated and unstyrenated alkyds in terms of viscosity and conversion. All the resins showed good properties. The styrenated alkyd resins gave a faster drying time and displayed better chemical resistance. Unlike the unstyrenated alkyd which is only resistant to water, brine, and acidic media, the styrenated alkyds were also resistant to alkalis. It was found that quadratic model equation best describes the dynamics of the physical drying and chemical resistance of both the styrenated and unstyrenated alkyds resins. Therefore, it can be concluded that styrenated alkyd resins offer an improved applications in terms of drying and chemical resistance in surface coating purposes.**

*Keywords:* **Styrenation, Polyesterification, Palm Kernel Oil, Modelling, Surface Coating.**

## 1. INTRODUCTION

Today the use of renewable and non-conventional raw materials for preparation of resins is one of the important areas which can be explored by the researchers. Oils from both the origins, vegetables and animals, are such raw materials which are widely used to modify variety of resins to achieve desirable properties. Alkyds are essentially short-branched polyesters formed by poly condensation of a dibasic acid and polyhydric alcohol in the presence of the glycerides oil or oil derived from fatty acids. The presence of the oil facilitates good pigment wetting properties and, when unsaturated allows coherent films to be formed on cure but properties like hardness, durability, colour and gloss retention are adversely affected (Panda, 2010).

The use of palm kernel oil has been reported to be suitable for the manufacturing of alkyd resin (Uzoh et al., 2016). Although it is non-drying oil, yet it has been proved to be suitable when highly unsaturated styrene is modified with it. The drying oils which are regarded as standards in surface coating industry are very scarce and expensive. However, doubts have been expressed about the sustained successful use of these drying oils (examples rubber seed oil, Linseed, Tung, Safflower Jatropha curcas etc) for the large scale production of alkyd resin. This is because of low percentages oil yield. Also their alkyds are prone to yellowing due to high unsaturation (Uzoh et al., 2016).

The use of styrene in alkyd resins modification has been reported by Ikhuoria et al. (2011) and Uzoh et al., (2016). They found that advantages obtained from the styrene – copolymerized alkyds are faster drying harder film, better water and chemical resistance than can be obtained with straight alkyd resins. However, the copolymers of styrene and various materials retain some of the sensitivity of polystyrene to certain hydrocarbon solvents. The tremendous production capacity for styrene, resulting from its extensive use in the synthetic rubber program during the last war, its relatively low cost, and very high degree of purity make it of definite interest for the surface –coating industry (Panda, 2010).

In design and process control, knowledge of some physical and chemical properties such as viscosity, density, iodine value and acid value are very essential. It is very imperative to monitor how these properties change during polymerisation reaction and how chemical changes in product and by-product affect the physical properties of the reaction mixture. In literature, many authors had reported the drying and chemical properties of alkyd resins using standard methods for testing but they did not use empirical equations to test run their findings.

Thus, the present study seeks to investigate the drying and chemical properties of unstyrenated and styrenated palm kernel based oil-modified alkyd resins using some empirical equations. In this study, different types of anhydrides of dibasic acid including aromatic and aliphatic category were used to prepare different types of alkyds and styrenated alkyds and their drying properties were analysed in detailed.

## 2. MATERIALS AND METHODS

### 2.1. Materials

Laboratory-grade phthalic anhydride with assay 99.7%, maleic anhydride with assay 99.0%, glycerol with assay 99.7%, styrene with assay 99.5%, xylene with assay 98%, benzoyl peroxide with assay 96%, and manganese (drier) from British Drug House (Poole, UK) were used in the preparation of the alkyds and styrenated alkyds. The palm kernel oil (PKO.) was purchased from Head Bridge Market, Onitsha, Anambra State in a refined form and used without further purifications. Distilled water was used throughout the analytical procedures.

### 2.2. Methods

#### 2.2.1. Preparation of Alkyd Resin

Three different alkyds were prepared with dehydrated palm kernel oil, glycerol, phthalic anhydride, maleic anhydride using lithium hydroxide oxide as catalyst using the formulations shown in Table 1. The reactions were carried out in a three necked round bottom flask titled with a motorized stirrer, a dean-stark trap titled with water-cooled condenser and nitrogen in let tube at a temperature of $230\text{-}250^0$C. Xylene was also employed as the azeotropic solvent. Two stages were involved

State 1 (Alcoholysis): At this stage, the measured quantity of dehydrated palm kernel oil was poured into the flask and heated to about $120^0$ to remove moisture. The heating was achieved with a heating mantle. Thereafter, the measured quantity of glycerol was added and the temperature was raised to $230^0$C. After 30 minutes, a small quantity of the aliquot was taken to check for its solubility in methanol. Alcholysis was completed when the solubility test in methanol was positive. The reaction mixtures was cooled to about $140^0$C

Stage 2 (Esterification process): At this stage, the measured quantity of phthalic anhydride, maleic anhydride and xylene was added into the flask and heated with a heating mantle. The temperature was gradually raised to about $230^0$C and maintained at a range of about $230\text{-}250^0$C for about 3hours. Aliquots were withdrawn from the reaction mixture at time intervals of 30minutes to check for drop in acid value. The reaction was then discontinued as soon as the acid value of the mixture attained the value of about 10mg K0H/g.

**Table 1: Recipe of the Preparation of Resin**

| Resin type | Oil length (%) | Glycerol (g) | Li0H (g) | PA (g) | MA (g) |
|------------|----------------|--------------|----------|--------|--------|
| Resin-A | 50 | 50 | 0.5 | 50 | - |
| Resin-B | 50 | 50 | 0.5 | 25 | 25 |
| Resin-C | 50 | 50 | 0.5 | 15 | 35 |

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### 2.2.2. Preparation of Styrenated Alkyd Resin

The styrenated alkyds were prepared by post co-polymerization of the alkyd resin. The alkyds were with-draw into 100ml flask and heated in the presence of an initiator, benzoyl peroxide, under reflux at $120^0C$ for 3hrs.

### 2.2.3. Drying Time Test

The prepared alkyd resin and styrenated resin mixed with drier were thinned with xylene and applied as thin panels. The samples were checked frequently to trace the dryness or tackiness by using the finger tip.

### 2.2.4. Chemical Resistance Test

The cured films of resins and styrenated resins were immersed in various chemicals of different concentrations like tetraoxosulphate (iv) acid ($0.1MH_2S0_4$), sodium hydroxide (0.1M Na0H), sodium chloride (5% solution) and distilled water for 24hrs at room temperature. After the time period, the weight loss was measured. The more the weight loss, the less the resins and styrenated resins are resistant to the respective chemicals.

### 2.2.5. Test for Empirical Model Equations

In order to obtain the best fitted empirical equations, eight kinds of equations (i.e linear, quadratic, logarithm and exponential) (Prapasad and Dutt, 1989) were tested using regression analysis method for each of the chemical (conversion) and physical properties (Viscosity) for each reaction, and the coefficient of determination ($r^2$) was determined from trend lines.

$$y = Ax + B \tag{1}$$

$$y = \log \left( A \left( \frac{1}{x} \right) + B \right) \tag{2}$$

$$\text{In} y = Ax + B \tag{3}$$

$$y = \exp \left( A \left( \frac{1}{x} \right) + B \right) \tag{4}$$

$$y = Ax^2 + Bx + C \tag{5}$$

$$\text{In } y = A \left( \frac{1}{x} \right)^2 + B \left( \frac{1}{x} \right) + c \tag{6}$$

$$\text{In } y = Ax^2 + Bx + C \tag{7}$$

$$y = A \left( \frac{1}{x} \right)^2 + B \left( \frac{1}{x} \right) + c \tag{8}$$

## 3. RESULTS AND DISCUSSION

### 3.1. Performance Characteristics of PKO alkyds and styrenated alkyds.

The performance characteristics of the alkyd resins and styrenated alkyd resin in terms of drying schedule and chemical resistance are shown in Table 2.

**Table 2: The performance characteristics of PKO alkyd and styrenated alkyd sample.**

| Sample | Drying schedule | | | Chemical resistance | | | |
|---|---|---|---|---|---|---|---|
| | STT (min) | SD (hr) | DT (hr) | 0.1M $H_2SO_4$ | 0.1M NaOH | 5% NaCL | Distilled $H_2O$ |
| Resin –A | 50 | 5 | 12 | 3 | 2 | 3 | 5 |
| Resin – B | 38 | 3.5 | 11 | 3 | 1 | 3 | 5 |
| Resin – C | 30 | 3 | 10.5 | 3 | 1 | 3 | 5 |
| SA | 25 | 2 | 5 | 4 | 4 | 4 | 4 |
| SB | 18 | 1.25 | 4 | 5 | 5 | 4 | 3 |
| SC | 15 | 1 | 3 | 5 | 5 | 4 | 3 |

**(1-5) = Rate of chemical resistance, STT = Set- to-Touch, SD = Surface dry, DT = Dry Through**

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The alkyd resins A, B and C were set-to-touch at 50, 38 and 30 minutes; surface dried at 5, 3.5, and 3 hours, and were completely dried at 12, 11 and 10.5 hours later. Their chemical resistance showed that they are resistant to acid, brine, and water but susceptible to alkali, as they were removed in alkali medium. This poor resistance to alkali occurs because ester groups of resins are readily attacked by alkalis and hydrolyzed by acid (Ikhuoria et al., 2011).

Their drying schedule of the styrenated alkyd resins as revealed in Table 2 shows an improvement from that of the pure alkyd resins, surface dry in 2, 1.25 and 1 hours; and dry through at 5 , 4 and 3 hours.

The chemical resistance of the styrenated alkyds is similar and also shows an improvement on the pure alkyd resins. Unlike the unstyrenanted alkyd which was only resistant to brine, water and acid, the styrenated alkyd resins also showed resistance to alkali. This means that styrenation assisted in making the ester linkages of the polyester from alkali hydrolysis (Ikhuoria et al., 2011).

### 3.2 Empirical Equations for resins

The test for empirical equations for alkyd resins has been successfully studied and their results are presented in Tables 3 and 4. The model equations were tested using viscosity and conversion for each of the reactions and the coefficient of determination ($R^2$) was determined by using analysis of regression.

**Table 3: Results on the test of empirical equations for s alkyd resins using viscosity and time**

| Components | DV | IV | Equation | Coefficients | | | $R^2$ |
|---|---|---|---|---|---|---|---|
| | | | | A | B | C | |
| Resin-A | Viscosity | time (min) | 1 | 0.033 | -0.84 | - | 0.888 |
| Resin-B | | | | 0.030 | 1.500 | - | 0.923 |
| Resin-C | | | | 0.035 | -o.246 | - | 0.988 |
| Resin-A | Viscosity | time | 2 | -63.46 | 1.621 | - | 0.816 |
| Resin-B | | | | -32.03 | 1.925 | - | 0.917 |
| Resin-C | | | | -43.36 | 1.678 | - | 0.935 |
| Resin-A | Viscosity | time | 3 | 0.013 | -0.678 | - | 0.937 |
| Resin-B | | | | 0.006 | 0.780 | - | 0.939 |
| Resin-C | | | | 0.010 | 0.019 | - | 0.969 |
| Resin-A | Viscosity | time | 4 | -137.2 | 4.569 | - | 0.605 |
| Resin-B | | | | -135.8 | 6.549 | - | 0.741 |
| Resin-C | | | | -137.4 | 5.304 | - | 0.769 |
| Resin-A | Viscosity | time | 5 | 0.000 | -0.012 | 1.01 | 0.990 |
| Resin-B | | | | $-7 \times 10^{-6}$ | 0.018 | 2.00 | 0.991 |
| Resin-C | | | | 0.000 | -0.024 | 2.12 | 0.994 |
| Resin-A | Viscosity | time | 6 | $6 \times 10^{-6}$ | -0.012 | -0.627 | 0.989 |
| Resin-B | | | | $7 \times 10^{-6}$ | 0.008 | 0.725 | 0.990 |
| Resin-C | | | | $4 \times 10^{-5}$ | 0.001 | 0.357 | 0.992 |
| Resin-A | Viscosity | time | 7 | 5042 | -262.3 | 2.938 | 0.932 |
| Resin-B | | | | 2449 | -128.6 | 2.564 | 0.974 |
| Resin-C | | | | 4521 | -221.7 | 2.859 | 0.993 |
| Resin-A | Viscosity | time | 8 | 15688 | -756.2 | 8.666 | 0.840 |
| Resin-B | | | | 12616 | -633.5 | 9.844 | 0.871 |
| Resin-C | | | | 17233 | -817.3 | 9.805 | 0.968 |

**Table 4: Results on the test of empirical equations for alkyd resins using conversion and time**

| Components | DV | IV | Equation | Coefficients | | | $R^2$ |
|---|---|---|---|---|---|---|---|
| | | | | A | B | C | |
| Resin-A | Conversion | time (min) | 1 | 0.003 | 0.405 | - | 0.834 |
| Resin-B | | | | 0.003 | 0.337 | - | 0.874 |
| Resin-C | | | | 0.003 | 0.388 | - | 0.944 |
| Resin-A | Conversion | time | 2 | -31.38 | 0.091 | - | 0.770 |
| Resin-B | | | | -31.13 | 0.044 | - | 0.899 |
| Resin-C | | | | -30.34 | 0.068 | - | 0.942 |
| Resin-A | Conversion | time | 3 | 0.005 | -0.865 | - | 0.743 |

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| Components | DV | IV | Equation | A | B | C | R² |
|---|---|---|---|---|---|---|---|
| Resin-B | | | | 0.005 | -0.964 | - | 0.813 |
| Resin-C | | | | 0.005 | -0.883 | - | 0.888 |
| Resin-A | Conversion | time | 4 | -18.88 | 1.00 | - | 0.670 |
| Resin-B | | | | -18.87 | 0.970 | - | 0.786 |
| Resin-C | | | | -18.65 | 0.990 | - | 0.831 |
| Resin-A | Conversion | time | 5 | $-3\times10^{-5}$ | 0.009 | 0.162 | 0.954 |
| Resin-B | | | | $-2\times10^{-5}$ | 0.007 | 0.179 | 0.972 |
| Resin-C | | | | $-3\times10^{-5}$ | 0.009 | 0.162 | 0.990 |
| Resin-A | Conversion | time | 6 | $-6\times10^{-5}$ | 0.017 | -1.356 | 0.870 |
| Resin-B | | | | $-4\times10^{-5}$ | 0.014 | -1.329 | 0.925 |
| Resin-C | | | | $-5\times10^{-5}$ | 0.0016 | -1.327 | 0.971 |
| Resin-A | Conversion | time | 7 | -212.8 | -22.99 | 0.036 | 0.945 |
| Resin-B | | | | 878.1 | -65.77 | 0.273 | 0.973 |
| Resin-C | | | | 398.0 | -46.05 | 0.172 | 0.986 |
| Resin-A | Conversion | time | 8 | 176.9 | -25.86 | 1.046 | 0.874 |
| Resin-B | | | | 851.2 | -52.45 | 1.192 | 0.971 |
| Resin-C | | | | 509.9 | -38.76 | 1.123 | 0.972 |

As shown in Table 3, the values of $R^2$ are highest in resin C, followed by resin B and least in resin A. This showed that the alkyd resin C dry faster than alkyd resins B and A. This could be as a result of high content of maleic acid in the resin C. With increase in the maleic acid content in resin, the degree of unsaturation increases which causes decrease in the curing time. The unsaturation is the main component for crosslinking reaction by free radical mechanism. Also, the values of $R^2$ are high enough for quadratic equations and least in linear equation. This could be explained by irregular change of viscosity with time. Quadratic equations are the best equation to describe the relation between viscosity and time, followed by logarithmic equation and least in linear equation respectively. This implies that the quadratic equations are the best equation to describe the drying properties of alky resins A, B, and C. Also in Table 4, the values of $R^2$ are highest in alkyd resin C followed by resin B and least in resin A. This equally showed that alkyd resin C dry faster than resin B and resin A. It was also observed based on the values of $R^2$ that the quadratic equations are the best equations to describe the relation between conversion and time. This means that quadratic equation (5) is the best equation to describe the drying properties of alkyd resins A, B and C in terms of conversion. In comparing viscosity and conversion, it was observed that conversion is the best parameter that can be used in those empirical equations to test for the drying properties of palm kernel oil-modified alkyd resins A, B and C and resin C gave the best drying properties because of high content of maleic acid present in it.

### 3.3. Empirical Equations for styrenated resins

**Table 5: Results on the test of empirical equations for styrenated alkyds using viscosity and time**

| Components | DV | IV | Equation | Coefficients | | | R² |
|---|---|---|---|---|---|---|---|
| | | | | A | B | C | |
| SA | Viscosity | time (min) | 1 | 0.117 | 4.205 | - | 0.988 |
| SB | | | | 0.098 | 4.445 | - | 0.995 |
| SC | | | | 0.092 | 2.183 | - | 0.996 |
| SA | Viscosity | time | 2 | -42.37 | 3.296 | - | 0.866 |
| SB | | | | -39.44 | 3.154 | - | 0.931 |
| SC | | | | -43.32 | 2.972 | - | 0.944 |
| SA | Viscosity | time | 3 | 0.007 | 1.891 | - | 0.965 |
| SB | | | | 0.008 | 1.484 | - | 0.990 |
| SC | | | | 0.007 | 1.842 | - | 0.997 |
| SA | Viscosity | time | 4 | -569.7 | 24.25 | - | 0.744 |
| SB | | | | -483.7 | 21.33 | - | 0.814 |
| SC | | | | -432.6 | 17.82 | - | 0.830 |
| SA | Viscosity | time | 5 | -0.000 | 0.139 | 3.302 | 0.995 |
| SB | | | | -0.000 | 0.143 | 2.642 | 0.997 |
| SC | | | | $2\times10^{-7}$ | 0.092 | 2.185 | 0.998 |
| SA | Viscosity | time | 6 | 2498 | -142.1 | 3.631 | 0.990 |
| SB | | | | 1433 | -99.16 | 3.677 | 0.998 |

| Components | DV | IV | Equation | A | B | C | $R^2$ |
|---|---|---|---|---|---|---|---|
| SC | | | | 1587 | -102.2 | 3.576 | 0.999 |
| SA | Viscosity | time | 7 | $-4 \times 10^{-5}$ | 0.016 | 1.543 | 0.989 |
| SB | | | | $-4 \times 10^{-5}$ | 0.016 | 1.486 | 0.995 |
| SC | | | | $-3 \times 10^{-5}$ | 0.015 | 1.213 | 0.996 |
| SA | Viscosity | time | 8 | 35128 | -1955 | 33.43 | 0.968 |
| SB | | | | 30333 | -1680 | 29.25 | 0.985 |
| SC | | | | 34846 | -1808 | 26.93 | 0.992 |

**Table 6: Results on the test of empirical equations for styrenated alkyds using conversion and time**

| Components | DV | IV | Equation | Coefficients | | | $R^2$ |
|---|---|---|---|---|---|---|---|
| | | | | A | B | C | |
| SA | Conversion | time | 1 | 0.004 | -0.180 | - | 0.898 |
| SB | | | | 0.005 | -0.402 | - | 0.965 |
| SC | | | | 0.004 | -0.266 | - | 0.969 |
| SA | Conversion | time | 2 | -248.2 | 3.160 | - | 0.959 |
| SB | | | | -349.9 | 3.043 | - | 0.983 |
| SC | | | | -222.3 | 3.161 | - | 0.994 |
| SA | Conversion | time | 3 | 0.019 | -3.879 | - | 0.839 |
| SB | | | | 0.022 | -4.681 | - | 0.848 |
| SC | | | | 0.019 | -3.896 | - | 0.925 |
| SA | Conversion | time | 4 | -1.385 | 0.327 | - | 0.823 |
| SB | | | | -1.468 | 0.214 | - | 0.847 |
| SC | | | | -1.509 | 0.288 | - | 0.881 |
| SA | Conversion | time | 5 | $-2 \times 10^{-5}$ | 0.008 | -0.372 | 0.964 |
| SB | | | | $6 \times 10^{-5}$ | 0.003 | -0.35 | 0.989 |
| SC | | | | $-2 \times 10^{-5}$ | 0.007 | -0.401 | 0.995 |
| SA | Conversion | time | 6 | $-1 \times 10^{-4}$ | 0.100 | -8.119 | 0.935 |
| SB | | | | $2 \times 10^{-5}$ | 0.016 | -4.330 | 0.979 |
| SC | | | | $-2 \times 10^{-4}$ | 0.068 | -6.498 | 0.992 |
| SA | Conversion | time | 7 | 72296 | -1570 | 5.942 | 0.991 |
| SB | | | | -27425 | 370.40 | -2.078 | 0.997 |
| SC | | | | -6144 | -83.76 | -0.101 | 0.998 |
| SA | Conversion | time | 8 | 114.4 | -40.57 | 0.695 | 0.992 |
| SB | | | | 142.9 | -50.40 | 0.669 | 0.995 |
| SC | | | | 113.7 | -40.44 | 0.651 | 0.997 |

**DV=Dependent variable, IV=Independent variable**

The number of the best equations and the model coefficients for styrenated resins are shown in Tables 5 and 6. The model equations were tested using physical (viscosity) and chemical (conversion) properties for each reaction, and the coefficient of determination ($R^2$) was determined by using analysis of regression. The results in Table 5 showed that the values of $R^2$ are highest in styrenated alkyd resin C followed by styrenated resin B and least in styrenated resin A. This showed that styrenated alkyd resin C dry faster than styrenated alkyd resins B and A.. Also the values of R² are high enough for quadratic equations and least for exponential equation (4). This implies that the drying characteristics of styrenated alkyd resins A, B and C could be best explained by quadratic model equations and equation (5) being the best equation to describe the relation between viscosity and time.

Also in Table 6, the values of $R^2$ are highest in styrenated alkyd resin C followed by styrenated resin B and least in styrenated resin A. This equally showed that styrenated resin C dry faster than styrenated resins B and A. It was also observed based on the values of R² that the quadratic equations are the best equations to describe the relation between conversion and time; and equation (5) being the best equation to describe the drying characteristics of styrenated alkyd resins A, B and C in which styrenated resin C dry faster than others. This could be as a result of high content of maleic acid in the resin C couple with the styrene content. With increase in the maleic acid content in resins as well as styrene content, the degree of unsaturation increases which causes decrease in the curing time. The unsaturation is the main

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component for crosslinking reaction by free radical mechanism. It was also observed from the Tables that some of the coefficients (i.e A, B, and C) are positive while some are negative. The positive coefficients show that viscosity, conversion and time increase in the same direction. This means that an increase in time will result in an increase in viscosity and conversion and so on.

In comparing viscosity and conversion of styrenated alkyds, it was observed from the Tables 5 and 6 that viscosity is the best parameter that can be used in those empirical equations to test for the drying characteristics of palm kernel oil-modified styrenated alkyd resins A, B, and C. Also when the drying properties of styrenated alkyds and unstyrenated alkyds are compared based on the values of $R^2$; it was observed from the Tables that the values of $R^2$ are highest in styrenated alkyd resins. This shows that copolymerization of alkyd resins with styrene has further improved their drying and chemical properties. The resultant styrenated alkyds dried much faster than pure alkyds and possessed better chemical resistance.

## 4. CONCLUSION

It has been shown in this research that post-polymerisation of alkyd resins with styrene has further improved the drying time and chemical resistance of alkyd resins. The resultant styrenated alkyd resins possessed better chemical resistance and gave faster drying time. The empirical equations tested for styrenated and unstyrenated alkyds showed that quadratic model equation(s) is the best model to describe both the drying and chemical characteristics of alkyds and styrenated alkyds. This study also showed that alkyd resin C with high percentage of maleic anhydride styrenated dried much faster than other alkyd resins and possessed best chemical resistance.

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