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Response Surface Methodology to Optimize the Vield of Alkyd Resin from Jatropha (Jatropha Curcas) and Sesame (Sesamum İndicum) Seed Oils Using CaCO3 as Catalyst

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Abstract: The low-cost feedstocks such as sesame (*sesamum indicum*) and jatropha (*jatropha curcas*) seed oils were utilized to optimize the yield of alkyd resins. The experimentally selected input factors ranges in the molar ratios of oil:glycerol (0.3 – 1), phythalic anhydride: glycerol (1 – 3), and catalyst (0.5–1.5 wt. %) for optimization were established using the response surface methodology (RSM) of Box Behken model to improve the alkyd resin yield factors. The optimization solution utilizing $CaCO_3$ catalysts, and a combination of other process factors evaluated, as well as the corresponding desirability functions, was found using analysis of variance (ANOVA) results for refined sesame alkyd resin (RSAR) and refined jatropha alkyd resin (RJAR). The RSAR optimization using a $CaCO_3$ concentration of 1.5 wt. % at a molar ratios of oil:glycerol (1.0:1.0) and phythalic anhydride:glycerol (3.0:1.0), while the RJAR at a similar catalyst concentration of 1.5 wt. %, molar ratio of oil:glycerol (1.0:1.0), and phythalic anhydride:glycrol (2.8:1.0) were observed for the alkyd resin optimization for the two processes. At these reaction conditions, the predicted and experimental biodiesel yield were 48.26 % and 47.29 % for RSAR and 62.07 % and 61.61 % for RJAR, respectively which shows less than 0.5% variations in both cases.

Key words: vegetable-oils, alkyd resin; optimization; jatropha; sesame

Katalizör Olarak CaCO3 Kullanan Jatropha (Jatropha Curcas) ve Susam (Sesamum İndicum) Tohum Yağlarından Alkid Reçinesinin Görünümünü Optimize Etmek için Tepki Yüzey Metodolojisi

Öz: Alkid reçinelerinin verimini optimize etmek için susam (sesamum indicum) ve jatropha (jatropha curcas) tohum yağları gibi düşük maliyetli hammaddeler kullanılmıştır. Optimizasyon için yağ:gliserol (0,3 – 1), fitalik anhidrit: gliserol (1 – 3) ve katalizörün (% 0,5–1,5 ağırlık) molar oranlarında deneysel olarak seçilen girdi faktörleri aralıkları yanıt yüzeyi metodolojisi kullanılarak oluşturulmuştur (Alkid reçine verim faktörlerini iyileştirmek için Box Behken modelinin RSM). CaCO3 katalizörlerini kullanan optimizasyon çözümü ve değerlendirilen diğer işlem faktörlerinin yanı sıra karşılık gelen arzu edilebilirlik fonksiyonlarının bir kombinasyonu, rafine susam alkid reçinesi (RSAR) ve rafine jatrofa alkid reçinesi (RJAR) için varyans analizi (ANOVA) sonuçları kullanılarak bulundu. . Ağırlıkça 1.5 CaCO3 konsantrasyonu kullanılarak RSAR optimizasyonu. yağ:gliserol (1.0:1.0) ve fitalik anhidrit:gliserol (3.0:1.0) molar oranlarında %, RJAR ise ağırlıkça 1.5 benzer bir katalizör konsantrasyonunda. İki işlem için alkid reçinesi optimizasyonu için %, yağ:gliserol (1.0:1.0) ve fitalik anhidrit:glikrol (2.8:1.0) molar oranı gözlendi. Bu reaksiyon koşullarında, öngörülen ve deneysel biyodizel verimi, RSAR için sırasıyla %48.26 ve %47.29 ve RJAR için %62.07 ve %61.61'dir ve bu, her iki durumda da %0.5'ten daha az varyasyon gösterir.

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1. Introduction:

Due to increased awareness of environmental concerns around the world, the use of renewable resources in various fields of polymer applications has proliferated day by day. Naturally, renewable resources have many advantages, such as feed availability, environmentally friendly nature, and low cost [1]. Nigeria's extensive forest resources and farmland yield a range of oil-bearing products sowing plants. For the manufacture of diverse polymeric resins, such as polyester, epoxy, polyurethane, amide polyester, etc several seed oils were employed. [2]. Main seed oil traditionally used to make such resins is linseed, castor, soybean, sunflower, safflower, tung, coconut, etc. These resins were used in the various fields of application like paint, coating, adhesives, and composite binder. Avocados are of commercial value and are cultivated worldwide in tropical Mediterranean climates. They have fleshy, green-skinned bodies, it can be shaped like a pear, an egg, or a sphere. The self-pollinating, avocado trees are propagated by grafting to preserve a projected quality and quantity of fruit [3,4].

According to Aigbodion, *et al.* [5,6], alkyds are important basic ingredients for making a variety of surface coatings because they act as binders.. In the surface-coating industry, vegetable oils are widely used in the production of oil-modified alkyd resin. The rising expense of traditional vegetable oils used in coatings, which contributed to price increases in finished coating products such as paints and lacquers, has led alkyd chemists to hunt for other, but less expensive, sources of surface-coating vegetable oils. [7]. The oil chosen for alkyd production appears to have a profound impact on the properties of the saturated bond amount presumably contributing to its non-dryness properties. Aigbodion and Okieimen [5], and Ikhuoria, *et al.* [8] studied the castor oil-modified alkyd resin production and characterization drying air for surface coatings.

Muturi-Nwangi, *et al.* [9], documented synthesis of alkyd resins using a partially and completely epoxidized vernonie oil, linseed oil, and soybean oil. The less yellowing property demonstrated the completely epoxidized linseed oil, and this yellowing of a vegetable oil could be managed by reducing the amount of unsaturation. Gogte and Sarwadekar [4] proved that alkyd resin prepared in conjunction with castor oil, from fractionated rice bran oil was of very good quality. The result showed that rice bran oil allowed for the use of maleic acid without the danger of the prepared resin being gelated. Igwe and Ogbobe [10], studied alkyd resin synthesis using melon seed, and oils from rubber seed. The results revealed that in the synthesis of alkyd resins, linseed may be replaced by rubber seed oil and soybean oils. Melon seed oil was shown to be a good substitute for linseed oil and soya bean oil in the synthesis of various alkyd resins.. The epoxidized soybean seed oil had greater potential for use in the formulation of surface coating based on drying movie resources. The alkyd resins derived from both altered and epoxidized soybeans modified alkyd soybean resins showed excellent chemical and alkaline resistance. By processes of alcoholysis-polyesterification Alkyd resins containing jojoba seed oil were created by Shaker et al. [11]. Also, Box *et al*. [12] developed Response Surface Methodology (RSM), which is based on computer simulations or experimental observations [13-15]. This study used response surface methods to provide the best analysis of factors on alkyd resin yields from jatropha and sesame seed oils.

2. Materials and Methods

2.1 Seed Collection and Extraction

The sesame and jatropha seeds were collected in farms and markets in Ilorin, Kwara State, Nigeria. A 40g of freeze dried raw crushed sample was placed in a beaker with 200 mL n-hexane and agitated at 300 rpm for 20 hours at 45 ^{o}C for n-hexane extraction. To prevent the solvent from evaporating, the lip of the beaker was securely closed with aluminum foil paper. The hexane solution was filtered with 125 mm grade filter paper after extraction and evaporated at 400°C in a rotary vacuum evaporator. Until examination, the extracted oil was kept at room temperature (20^{o}C).

2.2. Refinement of the crude oils

This was a crucial step in the manufacturing of alkyd resins because it allowed for the removal of contaminants like phosphorus compounds, which were necessary for successful trans-esterification.
Degumming, alkaline, and bleaching treatments were used in the refining process [16-18].

2.3. Two stage - alky resin preparation – alcoholysis and esterification

Alcoholysis stage

The reactor was charged with 2 grams of $CaCO_3$ (catalyst) and 50 grams of each oil. The mixture was then heated to roughly 120 ^{o}C. A 20g glycerol was added at 120 oC and heated to 230-250 ^{o}C (the alcoholysis stage high temperature was deliberate to break the vegetable oil to monoglyceride and diglycerides , which in turn was

further broken down to two monoglycerides before esterification) with vigorous agitation for 30 minutes to cause trans-esterification of triglyceride into a mixture of mono- and diglyceride oils. Alcoholysis was completed when a sample of the combination formed became soluble in 1 to 3 liters of anhydrous methanol, providing a clear solution. To ensure a seamless transition to the esterification stage, the reaction temperature was reduced to 140°C.

Esterification stage

After 30 minutes, the temperature was lowered to 150 oC, and 25g phthalic anhydride, then xylene, were added. To increase the resin's molecular weight, the esterification water is withdrawn at regular intervals before the temperature is raised to 245 ºC with stirring.. By heating the bulk to temperatures above 250 ºC and stirring continuously, water was extracted along with the unreacted acid. The 5 hour reaction procedure was monitored by monitoring the acid number and viscosity on a regular basis. When the resulting solution became viscous and the acid value dropped below 10, it was stopped. Titrating to the phenolphthalein end point (1:1) with a 0.1M KOH solution after dissolving in a mixture of toluene and ethanol was used to determine the acid value of in-process samples taken at intervals [19].

2.4 Optimization of seed oil-based alkyd resins

The Process optimization is necessary not only to ensure or detect the best reaction parameters for a better yield or intended response, but also to provide high-quality products with shorter drying times, acceptable color, low production costs, and so on. The amount of time it takes for the product to dry is crucial to its quality. Additionally, lowering the product's cost by reducing reaction time and temperature is constantly taken into account. The MRS optimization method will be used to optimize the process, and the criterion feature will be used to input the required value for obtaining a targeted response. [20].

2.5 Experimental designs for yield optimization

The design of experiments (DoE) was established using RSM with a design input to measure the outputs or response. The DoE input variables used are: glycerol/oil molar ratio (M_1), catalyst (C), temperature (T), and glycerol/phthalic molar ratio (M_2). The three factors and three levels of a Box-Behnken Design (BBD) was adopted Box and Behnken [21]. The selected experimental factors and their ranges for optimization are glycerol/phthalic molar ratio (1-3), reaction NaOH catalyst (0.5–1.5 wt. %), reaction temperature (200 – 260 ° C), and glycerol/oil molar ratio (0.3-1), while the pressure was kept constant. The expected measurable outputs are yield as shown in Table 1 contains the variable ranges to cover the intervals.

Table 1. Experimental design levels with independent variables

Inputs	Notation	Levels		
		-1	0	+1
Phthalic/Glycerol	Phyt/Gly	1	2	3
Catalyst	Cat	0.5	1	1.5
Oil/Glycerol	Oil/Glyc	0.3	0.65	1

3. Results and Discussion

3.1 Using response surface methodology to optimize alkyd resin yields.

A design matrix (inputs) must be created in order to measure the reaction of outputs; a Box-Behnkem design (BBD) with three variables and three levels was used. The following input factors were used to create the design of experiments (DoE): Oil-to-Glycerol ratio (Oil/Gly), Phthalic-to-Glycerol ratio (Phy/Gly), and C: Catalyst-to-Glycerol ratio (Phy/Gly) (Cat.) The following were the factors that were chosen for optimization ranges based on experiments: The factor ranges spanned the intervals that were typically used in the literatures: Oil/Glyc (0.3–1), Phyt/Gly (1–3), and catalyst (0.5–1.5 wt. %) [22].

3.1.1 Refined jatropha-based alkyd resin

In this case, 17 experiments were needed to convert the entire range of choices and choose the optimal alkyd resin manufacturing procedure. The design matrix displays the number of experiments as well as the associated values for each variable combination (Table 2).

Table 2. Design experimental matrix at different input variables

	Factor 1	Factor 2	Factor3	Response	
Run	A:Oil/Glyc	B:Phyt/Gly	C:Catg	Yield %	
				Actual	Predicted
1	1	2	1.5	55.8	56.64
2	0.65	2	1	22.4	22.40
3	0.65	2	1	22.4	22.40
4	0.3	1	1	5.15	4.94
5	0.65	2	1	22.4	22.40
6	0.65	3	0.5	43.2	42.42
7	0.3	2	0.5	14.65	13.81
8	0.65	2	1	22.4	22.40
9	0.65	3	1.5	46.6	45.55
10	1	3	1	58.7	58.91
11	0.65	1	1.5	30.48	31.26
12	0.65	1	0.5	25.69	26.74
13	1	1	1	50.55	48.93
14	0.3	2	1.5	18.37	17.80
15	0.3	3	1	23.29	24.91
16	0.65	2	1	22.4	22.40
17	1	2	0.5	52.4	52.97

Analysis of variance

These equations show the second order polynomial functions that were obtained to model yield

$$\text{Yield} = +31.47181 + 12.94745\text{oil/glyc} - 13.59679\text{phyt/gly} - 54.38536\text{cat} - 7.13571\text{oil/glyc*phyt/gly} - 0.457143\text{oil/glyc*cat} - 0.695000\text{phyt/gly*cat} + 44.22449\text{oil/glyc}^2 + 6.60500\text{phyt/gly}^2 + 29.95000\text{cat}^2 \quad (1)$$

The oil/glyc*phyt/gly, oil/glyc*cat, and phyt/gly*cat reflect the linear interaction effects between the refined jatropha oil/glycerol, phthalic/glycerol, and catalyst, respectively. The respective process variables' quadratic effects are are oil/glyc^2, phyt/gly^2, and cat^2.

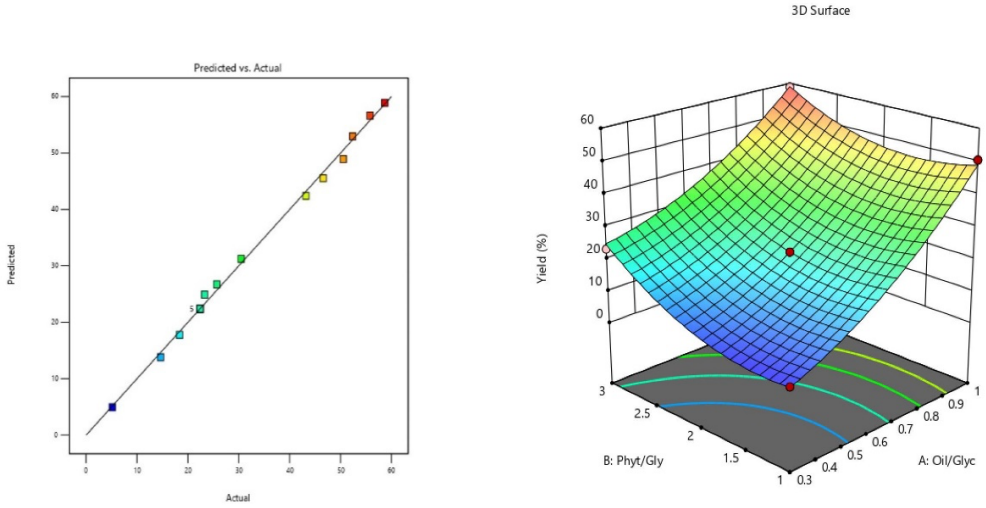
Table 3 shows how the regression equation was utilized to calculate the projected response.

It can be seen that all of the data points cluster together towards the straight y=x line. This indicates that the quadratic regression model developed was capable of accurately and confidently predicting the jatropha seed oil alkyd resin optimization process. As a result, this equation can be applied to both prediction and design.

Table 3. ANOVA Table for the "yield" quadratic model

Source	Sum of Squares	Df	Mean Square	F-value	p-value (prob > F)	
Model	4150.88	9	461.21	297.84	< 0.0001	significant
A-Oil/Glyc	3041.61	1	3041.61	1964.20	< 0.0001	
B-Phyt/Gly	448.80	1	448.80	289.82	< 0.0001	
C-Cat	29.30	1	29.30	18.92	0.0034	
AB	24.95	1	24.95	16.11	0.0051	
AC	0.0256	1	0.0256	0.0165	0.9013	
BC	0.4830	1	0.4830	0.3119	0.5939	
A^2	123.58	1	123.58	79.80	< 0.0001	
B^2	183.69	1	183.69	118.62	< 0.0001	
C^2	236.05	1	236.05	152.44	< 0.0001	
Residual	10.84	7	1.55			
Lack of Fit	10.84	3	3.61			
Pure Error	0.0000	4	0.0000			
Cor Total	4161.72	16				

The scatter diagrams show alignment of the predicted with the actual yields, viscosity and specific gravity as shown in Table 2 and Figure 1.



(a) (b)

Figure 1. Scatter diagram of (a) yield and (b) corresponding 3D surfaces

Table 4. Constraints

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Oil/Glyc	maximize	0.3	1	1	1	5
B:Phyt/Gly	maximize	1	3	1	1	3
C:Cat	maximize	0.5	1.5	1	1	3
Yield	maximize	5.15	58.7	1	1	3

Saponification, (mg/KOH)	148.42±0.27	182.55±0.2

Table 5. Optimization solution found according to RJAR optimization scenario

Number	Oil/Glyc	Phyt/Gly	Cat	Yield	Desirability	
1	1.000	2.621	1.500	62.066	0.932	Selected
2	1.000	2.628	1.500	62.162	0.932	
3	1.000	2.618	1.500	62.025	0.932	
4	1.000	2.642	1.500	62.345	0.932	
5	1.000	2.602	1.500	61.824	0.932	

Tables 2–4 illustrate the desirability functions for three separate criteria utilizing various input parameters (oil/glyc; phyt/gly; and $CaCO_3$ catalyst) and the combination of processes that were evaluated. Table 5 shows the optimization solutions discovered for the alkyd resin optimization scenario. The findings of the analysis of variance (ANOVA) revealed that catalyst dosages, oil/glycerol, and phthalic/glycerol molar ratios were critical factors in the formation of alkyd resin. The optimization solution yield was 62.066 %, with the selected overall desirability of 0.932, using the average input variables of catalyst (1.5 g), oil/glyc (1.0), and phyt/gly (2.621). Ikhuoria, *et al*. (2004), published studies on the enhancement of rubber seed oil methyl esters were used to make alkyd resins.. Methyl ester was more popular in rubber seed oil promising in alkyd resin growth, compared to rubber seed oil. Aghaie *et al.* [23] prepared alkyd resin with soybean oil fatty acid and compared the resin's properties with the resin made with other vegetable oils. The jathropa alkyd resin acid value decreased to 6.005±0.395 (mg/KOH), while alkyd resin viscosity improved to 10.88±0.042 (cp, 30 ºC) in the polymerization reaction to condensation after 5 hr. The production of soybean seed oil modified alkyd resins and epoxidized soybean oil was reported by Kyenge et al. [24]. The molecular weights increased from the jathropa oil (763.34 $gmol^{-1}$) to jathropa resin (1118.52 $gmol^{-1}$) with the saponification value of 148.42±0.27 (mg/KOH)

3.1.2 Refined sesame based alkyd resin

In this situation, 17 tests were also required to convert the complete range of options and select the best alkyd resin production method. The number of experiments and the related values for the combination of variables are shown in the design matrix (Table 6).

Table 6: Design experimental matrix at different input factors

Std	Run	Factor 1 A:Oil/Glyc	Factor 2 B:Phyt/Gly	Factor3 C:Cat g	Response Yield %	
					Actual	Predicted
8	1	1	2	1.5	50.55	51.96
14	2	0.65	2	1	38.55	38.55
13	3	0.65	2	1	38.55	38.55
1	4	0.3	1	1	4.35	9.34
15	5	0.65	2	1	38.55	38.55
10	6	0.65	3	0.5	24.9	30.60
5	7	0.3	2	0.5	16.21	14.80
17	8	0.65	2	1	38.55	38.55
12	9	0.65	3	1.5	25.29	28.88

4	10	1	3	1	59.285	54.29
11	11	0.65	1	1.5	28.52	22.82
9	12	0.65	1	0.5	24.68	21.09
2	13	1	1	1	45.25	49.55
7	14	0.3	2	1.5	9.415	10.12
3	15	0.3	3	1	24.47	20.17
16	16	0.65	2	1	38.55	38.55
6	17	1	2	0.5	47.995	47.29

Analysis of variance

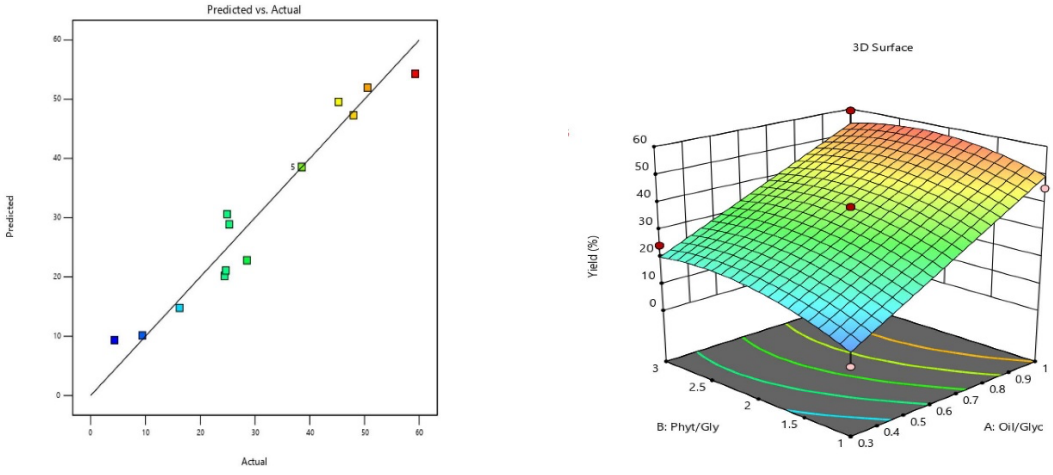
These equations show the second order polynomial functions that were obtained to model yield.
Yield = -54.99454 + 48.50587Oil/Glyc + 29.25580Phyt/Gly + 54.76036Cat - 4.34643Oil/Glyc*Phyt/Gly + 13.35714 Oil/Glyc*Cat - 1.72500Phyt/Gly*Cat - 0.066327Oil/Glyc^2 - 5.20312Phyt/Gly^2 - 29.99750Cat^2 (2)

The oil/glyc*phyt/gly, oil/glyc*cat, and phyt/gly*cat reflect the linear interaction effects between the refined jatropha oil/glycerol, phthalic/glycerol, and catalyst, respectively. The respective process variables' quadratic effects are oil/glyc^2, phyt/gly^2, and cat^2.
Table 7 shows how the regression equation was utilized to calculate the projected response. It can be seen that all of the data points cluster together towards the straight y=x line. This indicates that the quadratic regression model developed was capable of accurately and confidently predicting the sesame seed oil alkyd resin optimization process. As a result, this equation can be applied to both prediction and design

Table 7. ANOVA Table for the "yield" quadratic model

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	3288.15	9	365.35	14.01	0.0011	significant
A-Oil/Glyc	2761.55	1	2761.55	105.86	< 0.0001	
B-Phyt/Gly	121.25	1	121.25	4.65	0.0680	
C-Cat	0.0000	1	0.0000	4.792E-07	0.9995	
AB	9.26	1	9.26	0.3549	0.5701	
AC	21.86	1	21.86	0.8378	0.3905	
BC	2.98	1	2.98	0.1141	0.7455	
A^2	0.0003	1	0.0003	0.0000	0.9975	
B^2	113.99	1	113.99	4.37	0.0749	
C^2	236.80	1	236.80	9.08	0.0196	
Residual	182.60	7	26.09			
Lack of Fit	182.60	3	60.87			
Pure Error	0.0000	4	0.0000			
Cor Total	3470.75	16				

The scatter diagrams show alignment of the predicted with the actual yields, viscosity and specific gravity as shown in Table 6 and Figure 2.



(a) **(b)**

Figure 2. Scatter diagram: (a) yield and (b) with the corresponding 3D surface

Table 8: Constraints

Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
A:Oil/Glyc	maximize	0.3	1	1	1	3
B:Phyt/Gly	maximize	1	3	1	1	3
C:Cat	maximize	0.5	1.5	1	1	3

Table 8. Optimization solution found according to the RSAR optimization scenario

Number	Oil/Glyc	Phyt/Gly	Cat	Yield	Desirability	
1	1.000	3.000	1.500	48.264	0.909	Selected
2	1.000	3.000	1.500	48.265	0.909	
3	1.000	3.000	1.500	48.265	0.909	
4	1.000	3.000	1.495	48.388	0.908	
5	1.000	2.978	1.500	48.455	0.908	

Tables 6–8 illustrate the desirability functions for three separate criteria utilizing various input parameters (oil/glyc; phyt/gly; and CaCO3 catalyst) and the combination of processes that were evaluated. Table 9 shows the optimization solutions discovered for the alkyd resin optimization scenario.The findings of the analysis of variance (ANOVA) revealed that catalyst dosages, glycerol, and phythalic were critical factors in the formation of alkyd resin.

The optimization solution has alkyd resin yield of 48.264 % was obtained using the average input variables of catalyst (1.5 wt. %), oil/glyc (1.000), and phyt/gly (3.00), with the selected overall desirability of 0.909.

Table 9. Optimization solution found according to the RSAR optimization scenario

Sample	Oil/Glyc	Phyt/Gly	Cat	Yield	Desirability	
RSAR	1.000	3.000	1.5	48.264	0.909	Selected
RJAR	1.000	2.621	1.5	62.066	0.932	Selected

Tables 10 demonstrate the optimization findings obtained from Tables 5 and 9 utilizing $CaCO_3$ catalysts and a combination of other process variables investigated, as well as the related desirability functions. The RSAR

optimization used $CaCO_3$ concentration of 1.5 wt. % at molar ratio of Oil/Glyc (1.0:1.0) and Phyt/Glyc (3.0:1.0) were computed and obtained 48.264 % yield with a desirability of 0.909, while the RJAR used a similar $CaCO_3$

Saponification, (mg/KOH)	148.42±0.27	182.55±0.2

concentration of 1.5 wt. %, at molar ratio of Oil/Glyc (1.0/1.0) and Phyt/Glyc (2.621:1.0) to obtain 62.066 % yield with a desirability of 0.932. Average input factors such as catalyst concentration of 1.5 wt. %, molar ratio

of Oil/Glyc (1.0:1.0), and Phyt/Glyc (2.621:3.0) were observed during the alkyd resin optimization for the two procedures. The sesame alkyd resin acid value decreased to 8.53±0.12 (mg/KOH), while alkyd resin viscosity improved to 8.87±0.26 (cp, 30 ºC) in the polymerization reaction to condensation after 5 hr. The molecular weights increased from the sesame oil (768.71 $gmol^{-1}$) to sesame resin (915.41 $gmol^{-1}$) with the saponification, value of 182.55±0.2 (mg/KOH).

Ibrahim, *et al*. [25], previously reported an alkyd resin production range of 2.56–23.93 %. However, when compared to the results for ordinary alkyd resin production in the literature [26-35], the optimized experimental yields reported in this work were both greater. We were able to obtain a successful combination of input variables using the Box–Behnken Design Response Surface Methodology, which resulted in better yields and quality that were comparable to commercially available alkyd resins, resulting in the study's novel outcomes.

3.2. The Fourier Transform Infrared (FT-IR) analysis of alkyd resins

The presence of an absorption band at 3461.74 cm^{-1} in the RSAR FT-IR spectra (Figure 3 and 4) and Table 11 verifies the presence of hydroxyl group in the alkyd resin. A signal at 2920 cm^{-1} indicated the presence of a C-H stretching vibration of a sp^3 carbon or aliphatic compounds, whereas a signal at 1720.32 cm^{-1} confirmed the presence of a C=O carbonyl functional group of a carbonyl compound, and an absorption band at 1448.02 cm^{-1} indicated the presence of Sp^3 (CH_3 bend) hybridized carbon of aliphatic compounds. The presence of C-O stretching vibration is indicated by the emergence of a peak at 1071.24 cm^{-1}.

Table 10. The FT-IR analysis of alkyd resin from RSAR, and RJAR

Frequency (cm^{-1})	**Functional groups**	**Observed vibration bands/peaks (cm^{-1})**	
		RSAR	RJAR
3600 – 3200	O–H stretching vibration (alcohol)	3461.74	3518.86
29500 - 2840	C-H aliphatic stretching vibration.	2920.32	2926.65
1750 – 1720	C=O stretching (ester)	1720.32	1710.82
1465 – 1440	- CH_3 bend	1448.02	1448.02
1390 – 1365	O-CH_2 bend	1384.70	1372.03
1200 – 1020	C-O stretching vibration	1071.24	1134.56

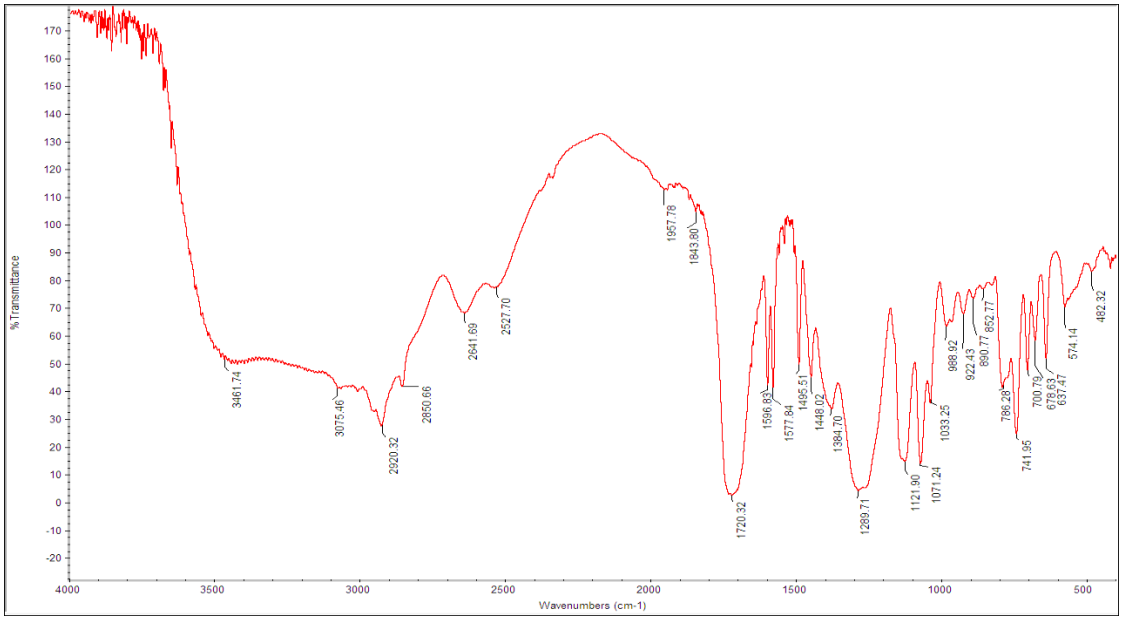


Figure 3. FTIR Spectra of the refined sesame based alkyd resin

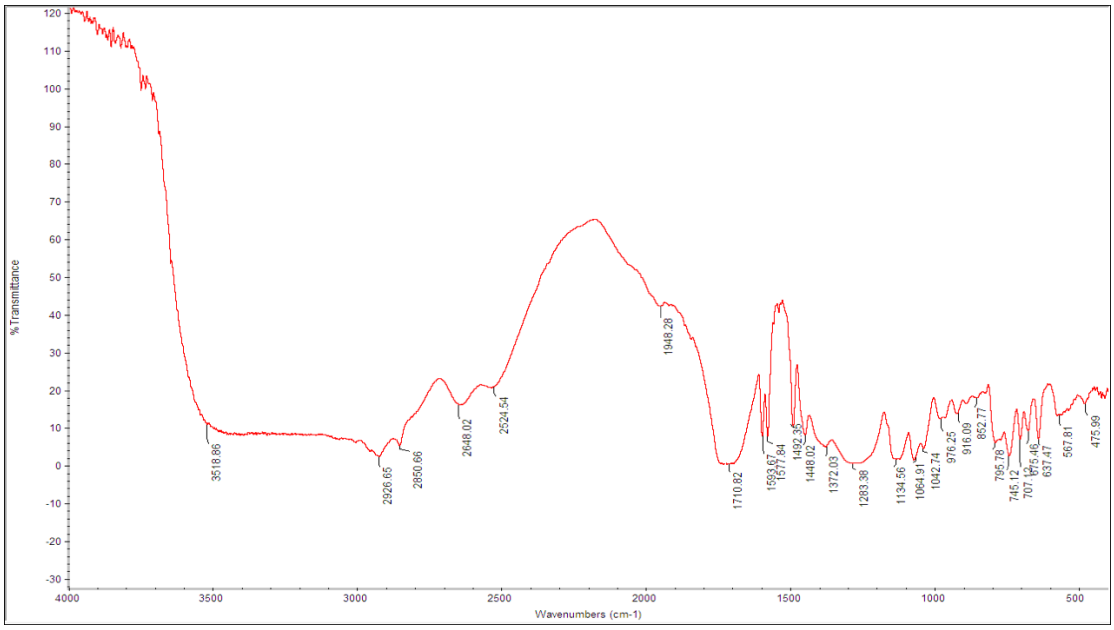


Figure 4. FTIR Spectra of the refined jatropha based alkyd resin

Table 11. Fatty acids profile of refined jatropha and sesame oils

	Refined Jathropa oil		Refined sesame oil		
Composition	Saturation	Composition (%)	Composition	Saturation	Composition (%)
Acetic Acid	$C_2H_4O_2$	1.92			
Octanoic Acid	$C_6H_{16}O_2$	4.22			
Oleic Acid	$C_9H_{12}O_2$	43.53			
n- Hexadecanoic Acid	$C_{16}H_{32}O_2$	18.09			
Octadecanoic Acid	$C_{18}H_{36}O_2$	8.76	Octadecanoic Acid	$C_{18}H_{36}O_2$	6.83
Dodecanoic acid, methyl ester	$C_{13}H_{26}O_2$	2.23			
Eicosanoic acid	$C_{20}H_{40}O_2$	1.62			
6-Octadecenoic acid	$C_{19}H_{36}O_2$	2.00	9, 17-Octadecadienoic acid	$C_{18}H_{32}O_2$	89.52
9- Octadecenoic acid,	$C_{19}H_{36}O_2$	1.61	9- Octadecenoic acid,	$C_{19}H_{36}O_2$	3.65

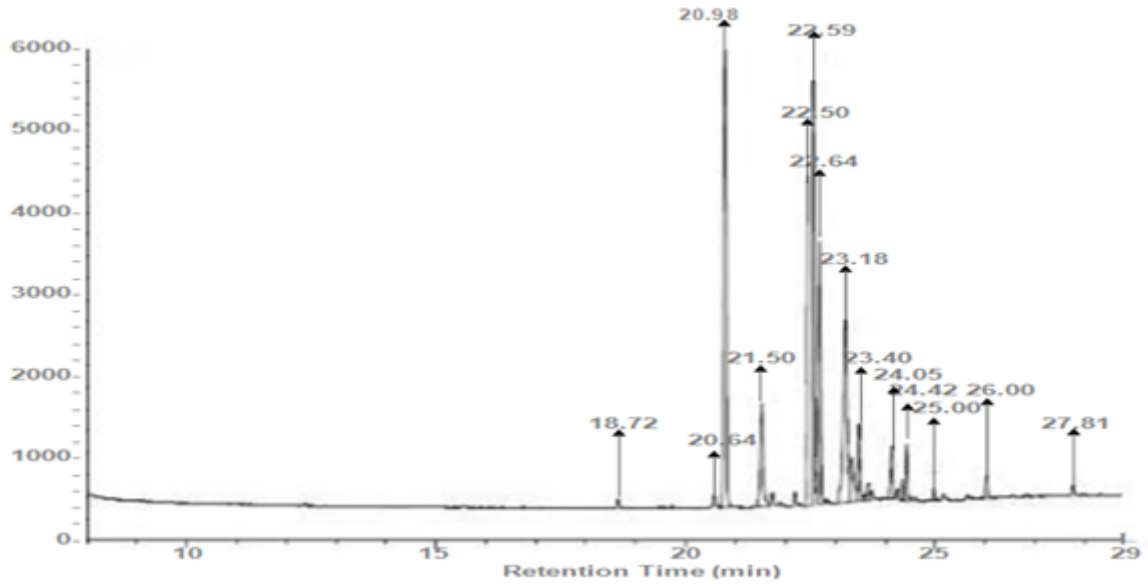


Figure 5. GC-MS Spectrum of refined jatropha oil

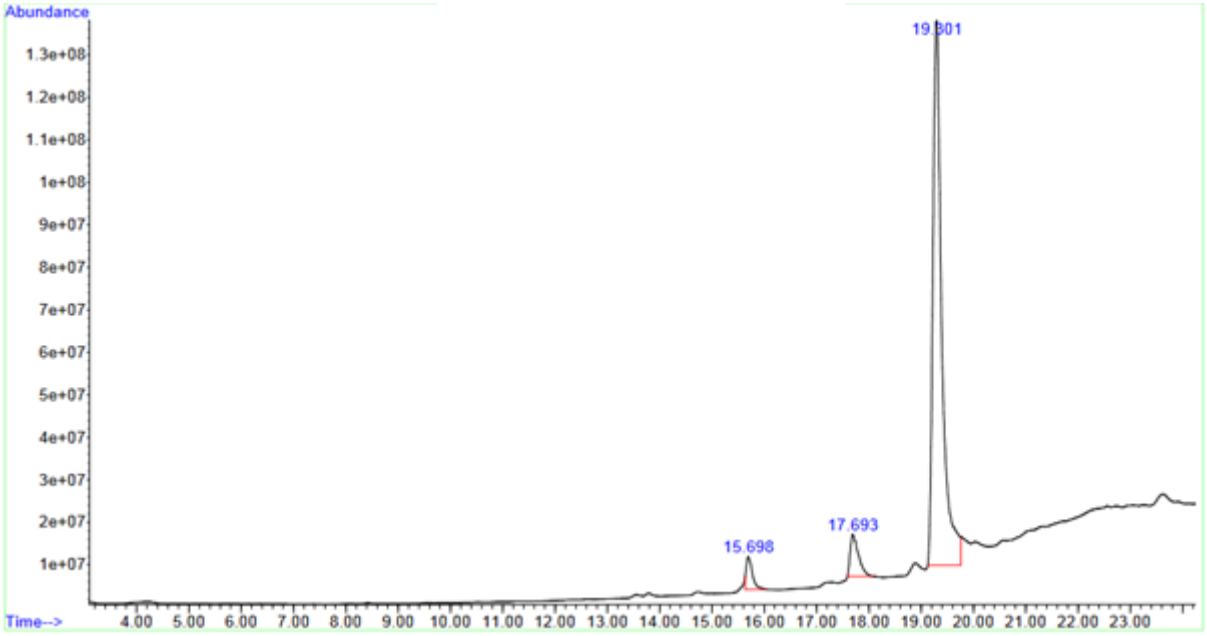


Figure 6. GC-MS Spectrum of refined jatropha oil

Figure 4 and Table 11 show the presence of an absorption band at 3518.86 cm^{-1} in RJAR's Fourier Transform Infrared (FTIR) spectra, indicating the presence of hydroxyl group in the alkyd resin. The appearance of a signal at 2926.65 cm^{-1} indicated the presence of C-H stretching vibration of a sp^3 carbon or aliphatic compounds, while the signal at 1710.82 cm^{-1} confirmed the presence of C=O carbonyl functional group of carbonyl compound, and the absorption band at 1448.02 cm^{-1} depicted the presence of sp^3 (CH_3 bend) hybridized carbon of aliphatic compounds. The development of a peak at 1134.24 cm^{-1} indicates the presence of C-O stretching vibration.

Table 12 contain the fatty acids profile of refined jatropha and sesame oils showed degree of unsaturation of different fatty acids with their corresponding gas chromatography spectral in figures 4 and 5, respectively. The oleic acid (43.53 %) and 9, 17-Octadecadienoic acid (89.52 %) observed as the most abundant.

5. Conclusion

The results from the ANOVA demonstrated that, although non edible seeds oil were different seeds, their molar ratio (oil/glycerol and glycerol/phthalic), speed, time and catalyst were vital factors that affects the yield of alkyd resin. Under these conditions, the highest alkyd resin yielded 62.066 % for RJAR and 48.263 % for RJAR, respectively. The experimental design for the alkyd resins that were produced resulted in increased yields and quality in industrial application.

The alkyd resin outputs generated from the two sets of combination variable tests were used to test the accuracy of the anticipated methodology. The optimized outputs of yield, viscosity, and Specific gravity quality data were gathered for these separate, diverse alkyd resins. The effects of calcium carbonate catalyst doses on the yield, viscosity, and specific gravity of alkyd resin produced from the RSAR and RJAR were major parameters that influenced the yield significantly, while the specific gravity and viscosity varied only marginally. The regression model supplied the multivariate analysis coefficients (R^2) as a variation of the mean, suggesting that the models were capable of good desirability.

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