

Kinetics of Castor Oil Alkyd Resin Polycondensation Reaction

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Abstract

An appropriate kinetic law that governs some important conditions of the reaction process for dehydrated castor monoglyceride modified alkyd resin has been developed. A kinetic experiment was conducted following a standard procedure. The classical third order conversion rate model was utilized in determining the rate parameters while the viscosity-conversion model suggested by the free-volume theory was applied for the viscosity kinetics studies. The kinetic model considered for this study adequately predicts the reaction progress even beyond the actual gelation point. The effects of the system parameters on both the predicted yields and the corresponding conversion rates were documented in a well-designed sampling space implemented by statistical screening optimization paradigm. The effects of system parameters on the reaction rates further investigated based on Arrhenius equation detect a heavy mass transfer resistance during the esterification process. A detailed analysis of the response reveals a deviation from linear first order kinetics and possible transition to second and higher order kinetics in the later stages of the esterification reaction.

Keywords: Polycondensation reaction; Kinetic model; Nonlinear fit; Free-volume theory; Arrhenius equation; Activation energy

Introduction

The uses of sustainable material in different fields of application of polymer have been proliferating day by day because of the increased worldwide awareness of environmental concerns and depletion of world oil pool. In the surface coating industries, oil-modified polyesters or alkyds dominate as the most widely used solvent-borne binder [1]. These fairly high molecular weight esters which are products of polycondensation reaction of polyol, diacid, and mono-acid (fatty acids) are very flexible and versatile and have been found to be compatible with other polymer systems [2]. The usual choice of fossil fuel has been criticized on the basis of its high environmental impact [3].

Dehydrated castor oil (DCO) has been found to be a sustainable material for alkyd resin synthesis in terms of its availability, biodegradability and renewability. Dehydrated castor oil modified alkyd resin has been developed keeping two criteria in mind; the resin met all the technical and industrial standards of durability, short drying time, resistance to chemical, etc., and, met all the ecologically relevant standards [4-6]. Castor oil has been classified as semi-drying oil and owe its value as raw materials for decorative and protective coatings to the ability to polymerize or “dry” after they have been applied to a surface to form tough, adherent, impervious, and abrasion resistance films. The advantages claimed in surface coating applications include excellent odor, good drying properties, more uniform polymer structure, and lack of after-yellowing. The dehydrated castor oil is non-yellowing oil and so can meet requirements of coating industries [7-11].

Many significant efforts have been made to increase alkyd resin production. Many researchers have attempted to search the different sources for alkyd resin preparation. Aigbodion et al. [12] studied enhancing the quality of alkyd resins using methyl esters of rubber seed oil in 2004. Hlaing and Oo [5] studied the manufacture of alkyd resin from dehydrated castor oil. In various reports, the possibility of attaining a gelation point which is a menace in alkyd resin production was emphasized.

The basic industrial batch reactor monitoring system depends on operator’s experience supported with on-line measurements of conversion and the end-use properties including molecular weight

and viscosity of cold product for predicting or estimating reaction progress and product quality in alkyd polymerization processes. This implies that the treatment of industrial alkyd kinetics prediction-estimation problem basically involves intensive laboratory testing and on-line reactor calibration effort which usually involves considerable experimental delay time. Thus the results obtained at any given time reflect the state of the reactor motion at certain time in the past and the measurements are said to be discrete -delayed. A Proper monitoring/control of the batch reactor and process design, therefore, requires both present time and time-ahead prediction of reaction progress/product quality. In this regard, the usual practice in the industry involves interpolation/extrapolation on log-log viscosity-conversion chart. This practice guides batch operators reasonably well to certain acceptable level of success (driven by operator’s experience) in industrial alkyd reactor monitoring.

In order to design an efficient batch reactor for alkyd resin production from castor oil, a suitable kinetic mechanism and reaction rate constants must be found. The processing technique adopted in this research involves the basic transesterification process. This is achieved through alcoholysis- esterification interface, popularly known as the monoglyceride process, in which the fear of increasing the production cost was alleviated through standard parameter optimization paradigm. In the scheme, the triglyceride oil was first converted to intermediate product (monoglyceride) which is subsequently esterified in presence of phthalic anhydride (PA) as illustrated in Figure 1. The detailed optimization procedure applied to the production of the oil modified alkyd resin has been reported in [13]. There seems to be scanty or no reported detailed kinetic report on the production process. In fact, the existing alkyd kinetics studies [14-16], report

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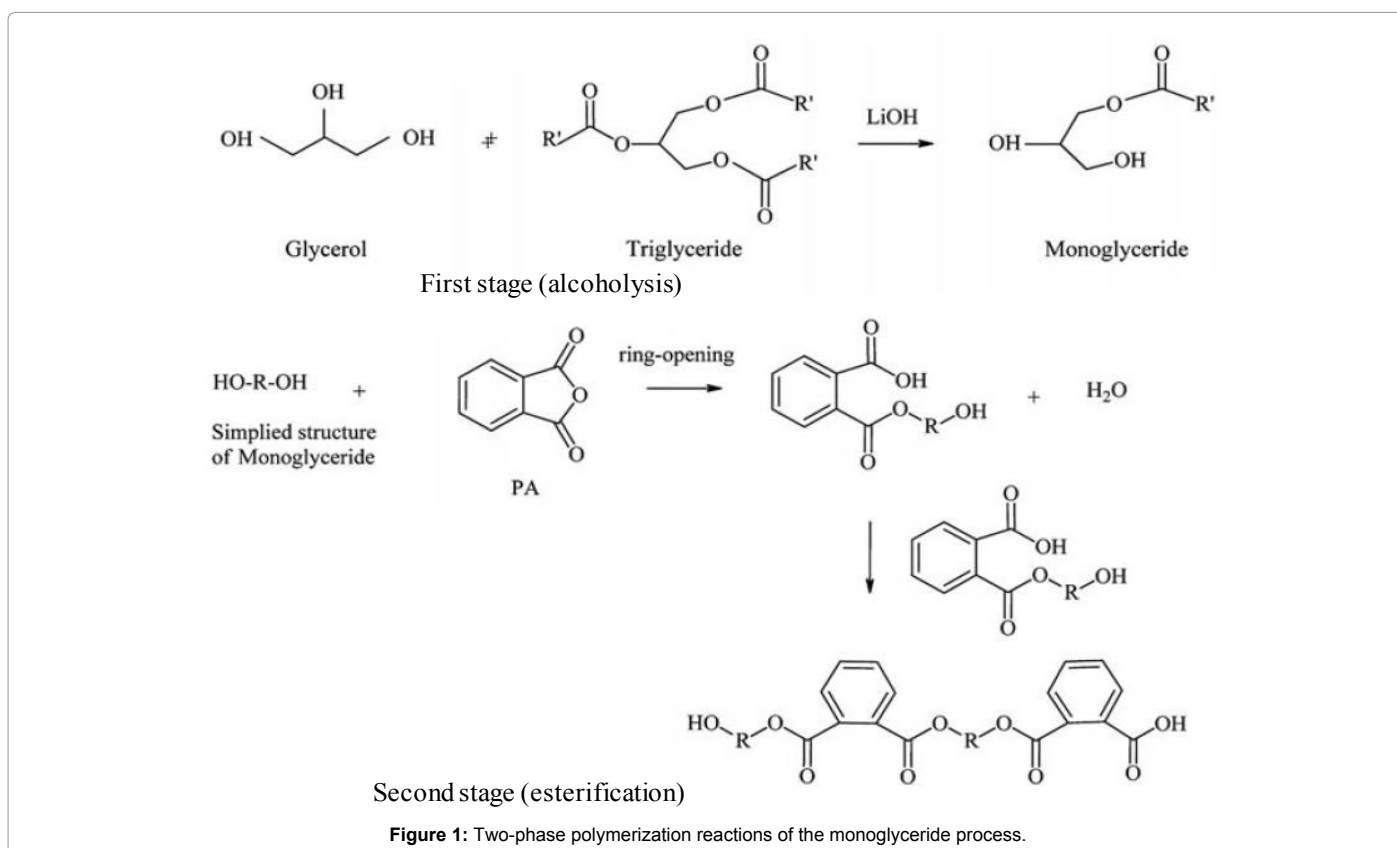


Figure 1: Two-phase polymerization reactions of the monoglyceride process.

simplified or lumped condensation-type kinetic models which are valid either at low or high conversion. Also, there is no reported literature on kinetics of dehydrated castor oil monoglyceride-based alkyd resin. The central focus of the current research, therefore, is to develop an appropriate kinetic law that governs some important conditions of the reaction process based on standard parameter optimization paradigm. A general predictive model was equally developed for the system responses (Y (acid value) and Y viscosity) as a major objective of the study, employing multivariate regression analysis conducted with respect to the identified system variables. The rate at which the process is executed is known to depend on the interactions of system parameters like reaction temperature (A), process duration (B), PA/oil ratio (C) and catalyst concentration (D). The multiple regression analysis sampling space is shown in Table 1, which depicts adequate range of the system parameters for process optimization as derived from a standard preplanning operation involving steepest ascent.

The proposed kinetics modeling adopts a simple but appropriate technique in which data obtained from the partial sampling procedure described by multivariate regression are fit to a reduced order reaction kinetic model. The reaction rate is related to the initial concentration of the reactants (a) and (b). The concentration of the mixture at later time (x) and the rate constant (k) are as follows;

$$\text{Rate } \frac{dx}{dt} = k(a-x)(b-x) \quad (1)$$

Also, the estimation of the unknown parameters of the generalized models involves some standard linearization techniques and the popular nonlinear least squares approximation algorithm. Subsequently, the resulting rate parameters were applied to the popular Arrhenius equation (2) through the nonlinear least square algorithm for evaluation of the thermodynamic variables which includes the

activation energy ΔE and frequency factor (A).

$$k = A \exp(-\Delta E / RT) \quad (2)$$

Materials and Methods

Materials

Castor oil was obtained in crude form from an oil mill at Nise, Anambra State, Nigeria. Equipment include; dean and stark apparatus, electronic weighing balance, heating mantle, magnetic stirrer and general laboratory glassware.

Phthalic anhydride ($C_6H_4(CO)_2O$) with minimum assay > 97%, glycerol ($C_3H_8O_3$) with assay > 99%, sodium bisulphate ($NaHSO_4$) with assay 97.5% and Lead(II) Oxide (PbO) with assay > 96.8% were purchased from BDH chemicals ltd, Poole, England. Diethyl ether, ethanol, xylene, anhydrous methanol, sodium hydroxide ($NaOH$) with assay 96%, was supplied by wharfedale laboratories, Yorkshire, England. The chemicals purchased were of highest purity.

Experimental

Preparation of alkyd resins was carried out with continuous azeotropic distillation of solvent which is conventionally 5wt% xylene of the total reactant and the reaction is set at a temperature from 220°C to 260°C with carbon dioxide purging for removal of water. Xylene is immiscible with water, as the mixture evaporates and collected in the Dean-Stark trap. Water is separated, while the upper layer, namely, xylene returns to the reactor system. Overall, solvent method offers better temperature and viscosity control. As there is no loss of raw material caused by volatilization and sublimation, it shows better control of the resin composition.

Independent Variable	Range and Level				
	- α	-1	0	1	α
Temperature (Celsius) (A)	220	230	240	250	260
Time (Minutes) (B)	30	60	90	120	150
Molar Ratio (C)	0.1:1	0.2:1	0.3:1	0.4:1	0.5:1
Catalyst Concentration (wt%)(D)	0.02	0.04	0.06	0.08	0.10

Table 1: Formulation of coded variables for experimental analysis.

Another difference in designing the reaction is the raw material choices of oils and fatty acid, which are usually called mono-glyceride process and fatty acid process, respectively. The mono-glyceride process is suitable for making alkyd with vegetable oils and glycerol. Heating those two components that has a mole ratio of 1:2 with a catalyst until the monoglyceride stage is achieved. After monoglyceride is formed, the dibasic acids and more polyols are charged into the flask. The reaction temperature was reduced to 220°C. After approximately 5 hours, which may vary according to the actual reaction temperature, the mixture became very viscous. The condensation reaction is stopped when the acid value is below 10 mg (KOH)/g. Castor monoglyceride (CM) modified alkyd resin was then prepared with the dehydrated oil, glycerol and phthalic anhydride using NaOH as catalyst.

In the synthesis of the alkyd resin, two stages were involved. The first stage was alcoholysis and the second stage esterification. The method suggested by Hlaing [5] for alkyd resin synthesis is utilized to implement a central composite rotatable design, formulated based on the factor setting indicated in Table 2. The coded notation of process variables was adopted because it enhances matrix transformation and result analysis. The details on the experimental design may be found in [13].

Alcoholysis: In this stage, Monoglyceride (MG) was first synthesized by reacting the dehydrated oil with glycerol. The oil was heated maintaining agitation speed of 700 rpm. Glycerol and selected catalyst (0.2% NaOH) was added and alcoholysis reaction was allowed to progress at 230-240°C. The reaction was continued until a sample of the reaction mixture became soluble in 2 to 4 volume of anhydrous methanol. After alcoholysis was completed, the reaction mixture was cooled to 14°C.

Esterification: At this stage, phthalic anhydride was added to the monoglyceride (MG) mixture. The temperature of the mixture was maintained at the range of 220 - 260°C. This second stage reaction is continued at constant temperature and long chain molecules were formed which contain excess hydroxyl group. At this state water was released. The mixed vapor generated was then condensed and collected. Xylene was suitably applied for this process considering its boiling point and solubility in water. The reaction progress was monitored by intermittent measurement of the acid value (AV) [14], viscosity and water off. The viscosity was measured following a standard procedure using Oswald viscometer. The data for the detailed kinetics study was gathered towards the end of the reaction when acid conversion approaches 87%. The acid value (AV) and viscosity were measured off-line for all reaction durations after a uniform delay period of 30minutes. The conversion of alkyd resin (Y) was calculated analytically in terms of measured reduction in AV (1) for a given reaction phase using equation 2. Data was obtained from normal titration while the viscosity was measured instrumentally for cold sample using viscometer.

The extent of reaction or conversion (Y) is estimated from the end group analysis of the aliquots of the reaction mixture withdrawn at 30 minutes interval.

$$\text{Acid value (AV)} = \frac{MV \times 40}{W} \quad (3)$$

M is the molarity of the solution (in moldm⁻³), W weight of the oil in (g) and V is volume of NaOH consumed

$$Y = 1 - \frac{AV_j}{AV_0} = 1 - \frac{V_j}{V_0} \quad (4)$$

AV₀ and AV_j are the acid values (in mg NaOH/g) of the mixture determined at the initial time (t=0) and later time t=j respectively while V₀ and V_j are the corresponding volumes of NaOH_(aq) used in the titration.

Kinetics modeling for the polycondensation reaction

i. The kinetic model used in this work relied on the following assumptions:

The esterification reaction was a reversible heterogeneous process, the rate of which was controlled by that of the chemical reaction.

ii. The chemical reaction occurred in the monoglyceride phase.

iii. There is excess -OH over -COOH.

iv. Monoglyceride concentration was assumed constant during the process.

Under these conditions, the reaction was assumed to be pseudo-homogeneous, first-order in the forward direction and second-order in the reverse direction, and hence conform to the following kinetic law



$$\frac{-dA}{dt} \equiv K_1^* [A] - K_2^* [C] \cdot [D] \quad (6)$$

Where;

[A] Denotes the concentration of acid in the reacting system (i.e. the acid value)

[C] and [D] are the concentrations of ester and water respectively, formed during the reaction.

K₁^{*} and K₂^{*} are the kinetic constants for the forward and reverse reactions respectively.

If [D] and [C] are assumed to be zero at the start (t = 0), and A = A₀ - E (E ≡ being the acidity removed), then, according to Carberry [15-17].

$$\frac{-dE}{dt} \equiv K_1(A_0 - E) - K_2 E^2 \quad (7)$$

A₀ is the initial acid number determined prior to the reaction.

If the forward reaction is of primary interest as indicated in the objective of the current research,

then, the second order term of the rate equation (7) may be ignored and the equivalent first order rate equation becomes;

PA/oil ratio	-2	-1	0	1	2
A	0.1672	0.2466	0.3576	0.3954	0.3353
ΔE(kJ/mol)	5.811	4.7564	3.4728	3.6449	3.7313

Table 2: Activation energies and frequency factors for castor oil modified alkyd resin synthesis.

$$\frac{-dE}{dt} \equiv K(A_0 - E) \quad (8)$$

On integration we have;

$$-\ln(A_0 - E) = -\ln(1 - Y) = K^*t + \gamma \quad (9)$$

Or

$$Y = 1 - e^{-(K^*t + \gamma)} \quad (10)$$

$$E = A_0 - A \text{ and } Y (\text{conversion}) = E / A_0 \quad (11)$$

The generalized first order kinetics constant K^* and the constant of integration γ were derived from the gradient and the intercepts of the linear plot of $-\ln(1 - Y)$ which corresponds to the forward process. The goodness of fit of the resulting model was tested numerically using the calculated norm of residuals ϵ_N . The general predictive model which was developed for the system responses ($Y_{\text{acid value}}$ and $Y_{\text{viscosity}}$) follows the method described in [13].

Conversion kinetics theory

One of the empirical relations applicable in the coating industry is the 3rd-order reaction rate equation of acid functional group conversion (12).

$$r = k(1 - c)(c_e - c)^2 \quad (12)$$

From standard kinetic modeling perspective, the reaction rate model (12) could be made to adequately describe the entire course of reaction by assuming a time varying reaction rate parameter as follows;

$$k(t) = \kappa(c(t), T(t)) \quad (13)$$

Which depends on variables like C and T .

If the batch can operate at constant temperature

$$\frac{dc}{dt} = r(c, k, c_e) = k(1 - c)(c_e - c)^2 \quad (14)$$

To simplify the integration of the third order conversion dynamics (14) and enhance estimation of rate parameters, an equivalent first order model is written (15).

$$\frac{dc}{dt} = r(c, k, c_e) = K^*(t)(1 - c) \quad (15)$$

From which the variables can be separated and integrated as follows;

$$\int \frac{dc}{1 - c} = \int K^*(t) dt \quad (16)$$

$$-\ln(1 - c) = K^*t + \gamma \quad (17)$$

$$\text{i.e.} \quad c = 1 - e^{-(K^*t + \gamma)} \quad (18)$$

$$\text{Hence} \quad Y = 1 - e^{-(K^*t + \gamma)}$$

The generalized first order kinetics constant K^* and the generalized integration constant γ can then be estimated from the gradient and the intercepts of the linear fit to the plot of $-\ln(1 - c)$ against time t . The adequacy of the linear fit may be monitored numerically using the statistically calculated norm of residuals ϵ_N or coefficient of determination R^2 .

Viscosity kinetics theory

The study of reactor kinetics based on viscosity measurement was carried out using viscosity - conversion relation suggested by the

classical free-volume theory [18]. The parameter estimation procedure was formulated in a non-linear least squares fashion. This approach requires initial guess of the model parameters. The unknown model parameters k , p_μ and c_e (kinetic parameter) are typically estimated for predicting the reaction progress and product quality in a batch reactor by fitting these calibration models to appropriate empirical data.

$$v = \mu(c, p_\mu) = a_\mu e^{b_\mu / (c_\mu - c)} \quad (19)$$

$$p_\mu = [a_\mu, b_\mu, c_\mu]'$$

Results and Discussion

The rate of a step polymerization is most conveniently expressed in terms of the concentration of reacting functional groups. Thus, the polyesterification was experimentally monitored by titrating for the unreacted carboxylic group with a base at every time step. Titration continues even after attaining a conversion of more than 87% towards the end of the polycondensation process. The results show that the identified variables were significant. The present kinetic study accounts for these major process parameters. Although the experiments were conducted in actual units of the variables, the result presentation and data analyses preferably adopt coded and uncoded notation for simplicity. A constant experimental delay period of 30 minutes was utilized, and at every time step, the conversion and cold viscosity of the emerging product were measured offline. The performances of the proposed reduced third order conversion model (15) and the free-volume type conversion-viscosity model (19) for time prediction of reaction progress and emerging product quality are tested for the pilot plant based on the experimental data.

Parameter estimation

The estimation of the rate constant k follows a linearization approach that results in equation (9) as described in section 2.3. The total allowed reaction period T considered to be zero (0) in the beginning of the monitoring was plotted against $-\ln(1 - c)$ as shown in Figures 2 and 3. The resulting gradient and intercept of the linear fit to the experimental data corresponds to the desired parameter estimates (11).

$$-\ln(1 - c) = K^*t + \gamma \equiv 0.00493t + 2.2688 \quad (20)$$

$$\text{Norm of residuals } \epsilon_N = 0.46089$$

Nonlinear least square fit

This is a relevant theory for constructing a mechanistic model which is nonlinear in the unknown parameters. It involves iterative procedure that starts with an initial guess of the unknown parameters. The subsequent iterations alter the current guess until the algorithm converges. This method also involves selecting the field function(s) or the mechanistic model(s) that would provide adequate fit to the experimental data.

The functions for fitting nonlinear models are of the form

$$y = f(X, \beta) + \epsilon \quad (21)$$

Where:

Y is an n -by-1 vector of observations

f is any function of X

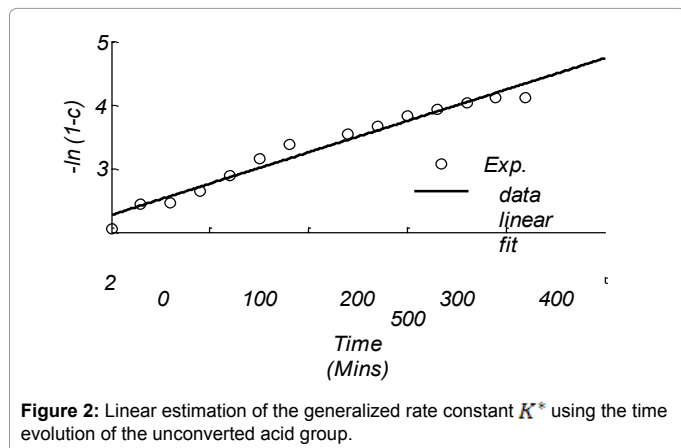


Figure 2: Linear estimation of the generalized rate constant K^* using the time evolution of the unconverted acid group.

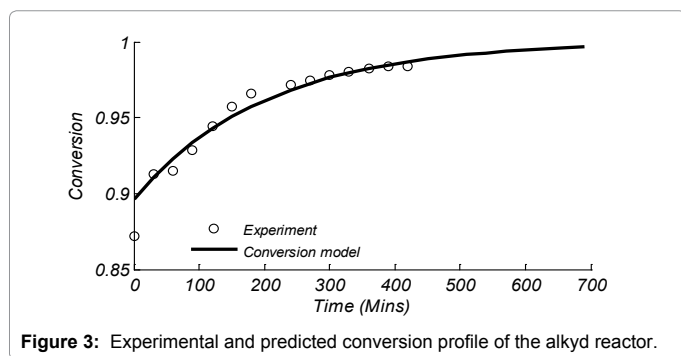


Figure 3: Experimental and predicted conversion profile of the alkyd reactor.

X is an n-by-p matrix of input variables

β is a p-by-1 vector of unknown parameters

Using the iterative method described, the unknown nonlinear parameters of the free-volume type

Conversion-viscosity model (19) was estimated as

$$p_{\mu} = [a_{\mu}, b_{\mu}, c_{\mu}]' = [8.4029, 0.568345]' \quad (22)$$

State estimation of conversion and viscosity

The results of the state estimation of the acid conversion presented in Figure 4 shows a good correspondence between the experimental data and those predicted by the proposed conversion kinetic model. The completion of the esterification process at which the operation of the batch may be stopped is established as the conversion value gets sufficiently close to 1 (i.e. 100%). This conforms to a total monitoring period greater than 300 minutes. However, the batch operation cannot be stopped without adequate consent to other product quality indicators like viscosity and gelation.

The viscosity profile of the alkyd reactor presented in Figure 4 also shows substantial agreement between the experimental and the predicted data. An important observation recorded in this figure is the sustained increase in the viscosity of emerging product as the cumulative delay period increase which emphasizes the need to mark out a critical gel point about which the operation of the batch must be stopped to ensure quality product. Nevertheless, selecting what is desirable in terms viscosity may vary depending on certain established standards and professional recommendation. The application of viscosity-conversion model proposed in this study for reactor monitoring guarantees one great advantage related with cost reduction on laboratory analysis since the on-line conversion measurement could be eliminated.

Effects of process parameters

Equations (23-24) [13] and Figures 5-8 disclose the predictive models for the responses $Y_{conversion}$ and $Y_{viscosity}$ and the detailed effects of the system parameters on the rate of conversion analyzed in a partial sampling space presented in Table 1.

$$Y_{conversion} = 79.82 + 7.64A + 10.14B + 5.42C + 5.20D - 3.79AB - 2.99BD - 3.85A^2 - 2.19B^2 - 2.82C^2 \quad (20)$$

$$Y_{viscosity} = 213 + 35.80A + 38.29B + 15.84C + 22.28D - 16.84AB - 16.39A^2 - 10.39B^2 - 9.31C^2 \quad (21)$$

The study was approached by investigating the fall in AV (conversion) as a function of time within some economically viable settings of the other independent system variables. As expected for increased degree of unsaturation, it was observed that the rate of forward reactions were generally improved by slight upward adjustment of the PA/oil ratio up to $C = 1$ while the reaction temperature (A) and catalyst concentration (D) assumed some fixed values. This observation was recorded in the linear rate plot of the experimental data presented in Figures 9-11. It appears that the rate of reaction of the polybasic acid (PA) with the primary hydroxyl groups of the monoglyceride to form linear polyesters was favoured at the stated condition. This trend however reversed at $C > 1$ due to rapid depletion of the primary hydroxyl group in excess of the acid which equally introduced curvature in the reaction path.

The study assumed that the forward reactions were satisfactorily described by a linear first order kinetic law of the general form

$$r = K^* t + \gamma \quad (25)$$

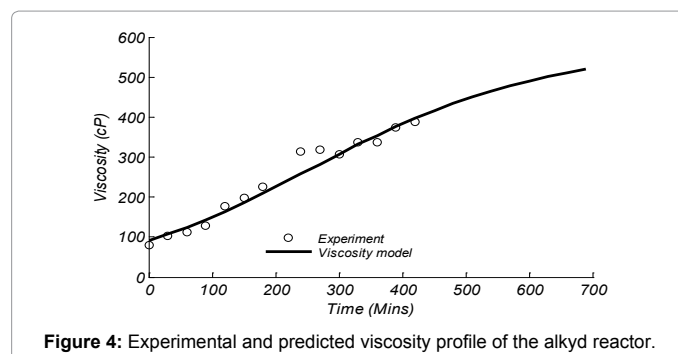


Figure 4: Experimental and predicted viscosity profile of the alkyd reactor.

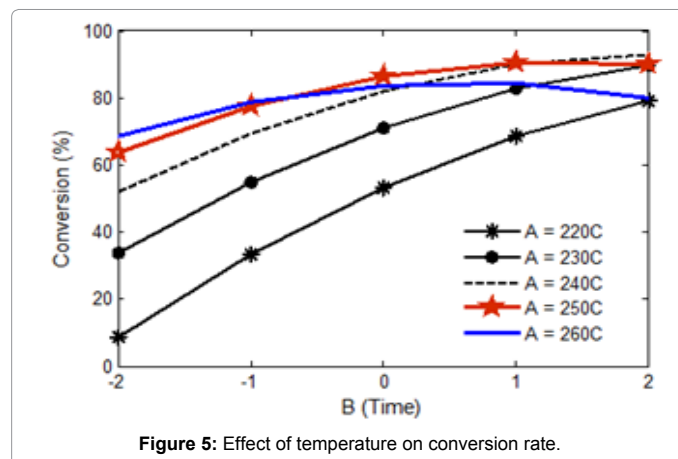
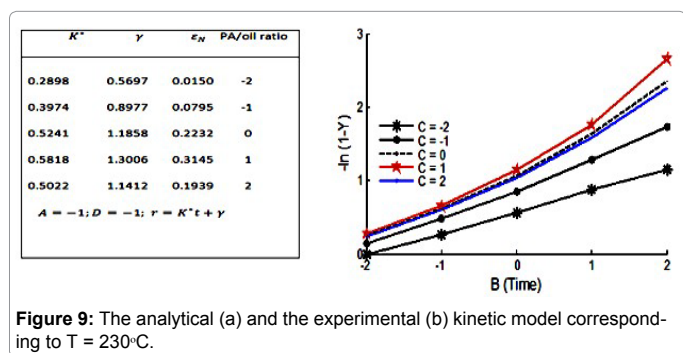
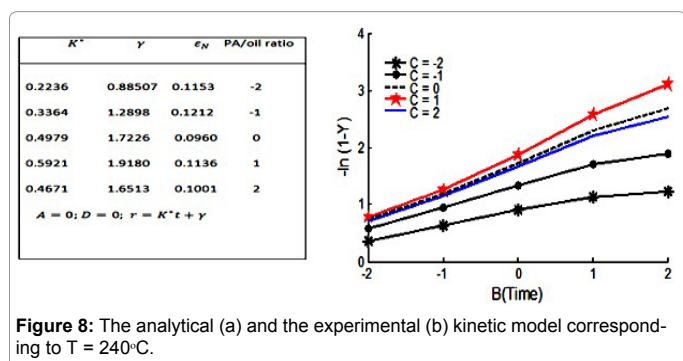
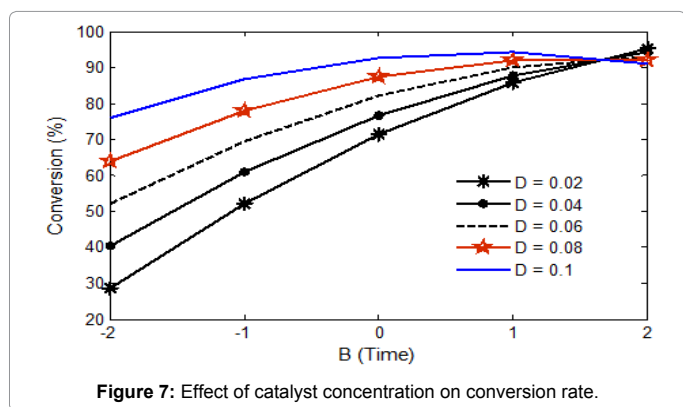
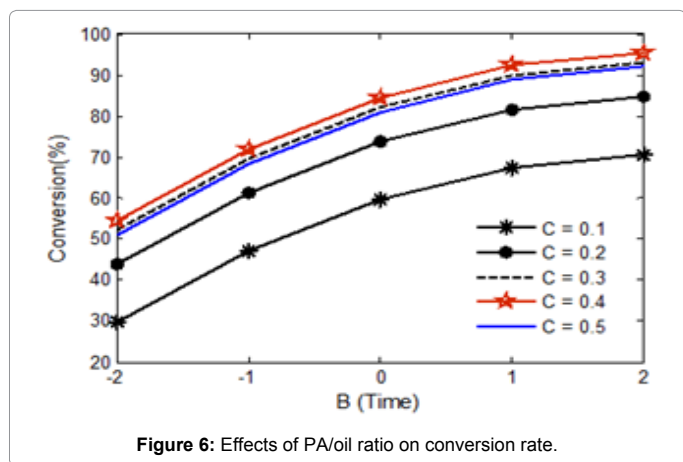


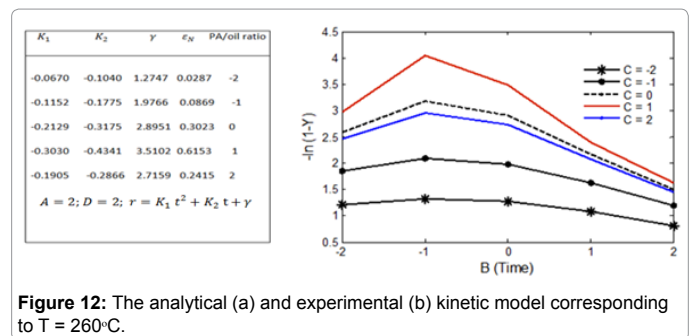
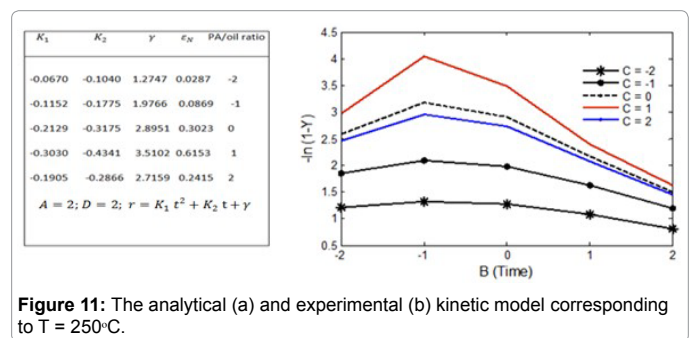
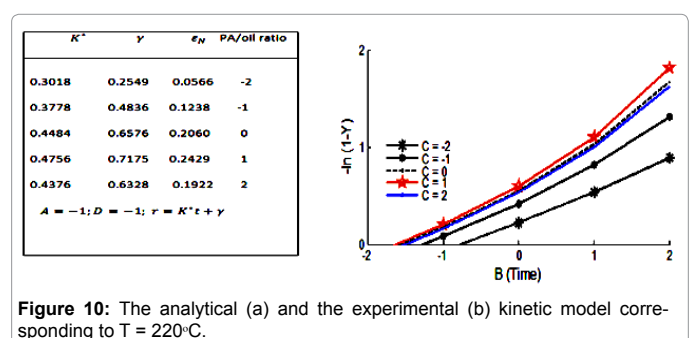
Figure 5: Effect of temperature on conversion rate.



This was particularly in the lower isotherms. The validity of the linear rate model within the optimum parameter range was however tested by evaluating the values of the coefficients K^* and γ of the rate

equation such that an acceptable goodness of fit verified numerically with calculated norm of residuals (ϵ_N) is obtained between the result of (25) and the experimental data. The results presented in Figures 8-10 consistently returned low values of (ϵ_N) for each investigation step confirming good fit in the applicable space. An important observation was the evolution of curvature in the reaction path towards the end of the forward process, which equally suggests a negative shift in the equilibrium position. Detailed study of the curvature in the present research shows that non optimal local inflexion points are possible at much higher isotherms ($> 240^\circ\text{C}$). This equally explains the deviation from the more constrained linear type kinetic model to a robust quadratic model at elevated temperatures illustrated in Figures 12 and 13. The decline in response noticed after the optimum reaction time has been explained on the concept of the secondary hydroxyl group which normally comes to play at the latter stage while the primary hydroxyl is varnishing. Molecules of PA possibly react with the secondary hydroxyls and connect the short chains to form complex, branched or network structures and in effect impedes the rate of forward process [18].

The effects of PA/oil ratio on reaction rate was further investigated using the thermodynamic variables of the system supplied by the Arrhenius equation (2). The values of the frequency factor (A) and the energy term (ΔE) presented in Table 2 were attained via non-linear



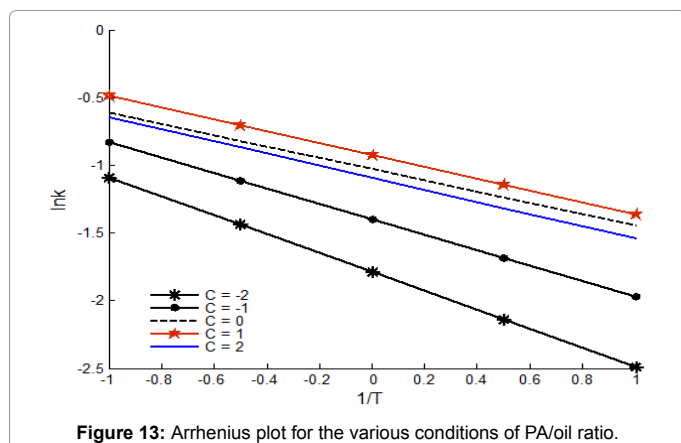


Figure 13: Arrhenius plot for the various conditions of PA/oil ratio.

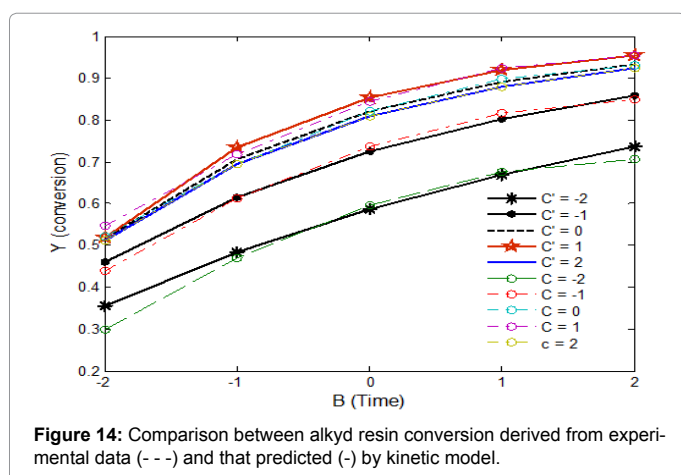


Figure 14: Comparison between alkyd resin conversion derived from experimental data (- - -) and that predicted (-) by kinetic model.

regression implemented on the plots of $\ln k$ against T^{-1} for various PA/oil ratios shown in Figure 13.

The validity of the linear type kinetic model in the forward process of esterification reaction is further demonstrated by comparing the alkyd resin conversions (Y) obtained with equation (23) derived from the fitted kinetics model and that obtained from equation (1) derived directly from experimental data, keeping the effects of temperature and catalyst at mean coded values. The comparison presented in Figure 14 shows a good correspondence between the results generated from kinetic model (presented in continuous lines) and that deduced from experimental data plotted in broken lines.

Conclusion

The kinetics of dehydrated castor oil modified alkyd resin has been studied within reasonable assumption. The results show that the forward reaction which favors the production of alkyd resin conform at large to the first order kinetic law while the reverse process is evidently second order. The rate at which the acid value of the reacting mixture drops in the course of reaction is shown to follow linear curves with approximately uniform gradient within the optimum isotherm in the first 120 minutes of polyesterification process. This trend was subsequently opposed at the later stages of the reactions due to fast degradation of the primary hydroxyl group and the uprising of the secondary hydroxyl group. The values of activation energy further detect a heavy mass transfer resistance during the esterification process. Overall, the present study on the kinetics of castor oil modified alkyd

resin synthesis lead to the following deductions which are important in the batch progress monitoring as a cleaner technology:

- The uncertain kinetic model used led to an on-line estimation of reaction rate parameter and the derivation of the proposed rate model,
- The proposed reduced third-order reaction rate model is adequate for predicting the overall acid functional group conversion of the alkyd reactor for the end-use properties
- The on-line monitoring of the alkyd reactor based on a viscosity measurement and conversion sufficiently guarantees optimal operation of the batch.

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