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Characterization of Alkyd Resins Prepared from Okra Seeds Oil with Two Polybasic Acids

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Abstract

The iodine values of Clemson spineless, Ex-samaru and NHAE-74 have been reported to be within the range of semi-drying oils. Hence, the oils can be used as raw material in coating industries. This study aimed to prepare alkyd resins from the oils of Clemson spineless, Ex-samaru and NHAE-74 using two polybasic acids (phthalic and maleic anhydride) with varying concentrations of maleic acids. The physical performance, chemical resistance, solubility and the viscosity of the alkyd resins were determined. The functional groups of the alkyd resin were also determined through Fourier Transform Infrared (FTIR) Spectroscopy. One way ANOVA was used to test the significance level of drying time and solubility in different solvents. The adhesion, abrasion, and chemical resistance tests of the alkyd resin were also evaluated. There was no significance difference ($p>0.05$) in the drying time for all the alkyd resins synthesized. The alkyd resins were resistant to distilled water and 5% $NaCl_{(aq)}$ but were fairly affected by 0.1 mol dm^{-3} H_2SO_4 (indicated by colour change) and dissolved completely by 0.1 mol dm^{-3} $NaOH_{(aq)}$. The alkyd resins were more soluble in polar solvents (acetone, ethanol and methanol) than non-polar (benzene and xylene) organic solvents and show excellent resistance to the adhesion and abrasion tests. Analysis of all the alkyd resins showed good performance characteristics, good resistance to distilled water and 5 % sodium chloride solution and fair resistance to the attack by sulphuric acid. The viscosity of the alkyd resins is proportional to concentration of maleic anhydride. Analysis of all the alkyd resins showed good performance characteristics, good resistance to distilled water and 5% sodium chloride solution, fair resistance to the attack of sulphuric acid, all the alkyds show more solubility in protic (acetone, methanol and ethanol) solvents than aprotic (benzene and xylene) solvents.It is recommended that, further research should be carried out with view of recommending best formulations of paints and varnishes with the produced alkyd resins with view of finding out the possibility of reducing the importation of alkyd resins in paints industries in Nigeria.

Keywords: Alkyd Resin, semi drying Oil, Phthalic anhydride, maleic anhydride, and wetting

1. Introduction

The rapid increase in human population in Nigeria has resulted to increase of house constructions and consequently marked increase in the demand for coatings and paints [1]. Nwuzor *et al.* [2] associated the high importation of alkyd resins into Nigeria to the lack of proper accessibility of local seed oils that can be utilized for their production. However, the potentials of many oil seeds for production of alkyd resins in Nigeria have not been investigated; hence, remain un-utilized for long period of time [3]. It is therefore, important to search for oils of such un-utilized seeds and investigate their possible applications in the field of alkyd resin production. Seeds oils (drying or semi drying oils) are used in production of alkyd resins which are used for the production of surface coatings such as decorative paints, industrial paints, air drying printing inks, stoving and varnishes [4]. It is reported that, use of dead fruit and bean seed oils for alkyd resin synthesis established new era that use locally sources of oils for structural, modern, industrial and architectural coatings which result in the increase in Nigerian economy. The plant, okra (*Abelmoschus esculentus* L), is an easy-to-cultivate indigenous crop [6]. The seeds have high oil yield (20-40%) and are rich in unsaturated fatty acids (60 to 70%) [7-8]. Abba *et al.* [9] determined the iodine value of various okra seed oils (110.97 – 114.95 $mgI_2/100g$) and classified them as semi drying oils. The iodine value is a key metric for determining the worth of oil as a raw material for alkyd synthesis [10]. Furthermore, compared to oil-based alkyd, the petroleum-based product has long term environment impact [11]. According to Uzoh *et al.* [12], oil-based alkyd coatings are easy to apply on poorly treated surfaces and provide strong corrosion protection, high gloss, and fast drying properties.

There has been tremendous increase in the demand for alkyd resin production for use in coating industries in Nigeria due to the construction of modern buildings in cities. Consequently, large quantity of oil is needed for the production of alkyd resin. Hence, there is need to exploit unconventional seeds oil with comparable properties with oils used in alkyd resin production and paint formulation.

The study aimed to synthesize alkyd resins from three varieties of okra (Clemson spineless, Ex-samaru and NHAE-74) seeds oil. The properties such as drying time, Adhesion, Abrasion, Chemical resistance and Solubility of the synthesized resins were investigated. FTIR and viscosity measurements were also used to characterized the synthesized resins.

The three different okra (*Abelmoschus esculentus*) seed varieties (Clemson spineless, Ex-samaru and NHAE-74) used in this research were obtained from the Institute of Agricultural Research, Ahmadu Bello University, Zaria-Nigeria. The dried okra seeds were further sun-dried for one week and ground to powder using a porcelain mortar [13]. Each variety's powder was sieved to remove seed's protective coat and packaged in a polyethylene bag for oil extraction.

2. Materials and Methods

2.2 Synthesis of Alkyd Resin

The oil was extracted from the seeds of three okra varieties using the method of Bouanga-Kalou *et al.* [14]. The extracted oil from each okra seeds was divided into three portions of 30 cm^3 each. Each portion was transferred into a three necked round bottom flask fitted with a motorized stirrer, a dean-stark trap fitted with water-cooling condenser, and a nitrogen in-let tube. Then 60 cm^3 of glycerol and 2 g of $Ca(OH)_2$ (catalyst) were added to the flask and heated at 200 ºC for 2 hrs. Alcoholysis was completed when a clear solution was obtained after mixing the portion of the hydrolyzed with methanol (ratio 1:1). The mixtures were cooled to about 180 ºC and 2 g of phthalic anhydride are added to each portion. Then, 0.0, 0.5 and 1.0 g of maleic anhydride were added to the portions 1, 2 and 3 respectively. Finally, 9 cm^3 of xylene was added to each reaction mixture to produce alkyd resin at a temperature of 220 ^0C till the end of the reaction [15 and 16]. The alkyd resin is allowed to cool to a temperature of 150 ºC, diluted with a mixture of toluene and ethanol (1:1) thoroughly to achieve homogeneity [18]. The above procedure was repeated with different maleic anhydride composition.

2.3 Performance Characteristic Tests of Alkyd Resin

2.3.1 Preparation of Surface Material

The surface material used (glass plate of 50 cm X 50 cm X 1 cm) was prepared as described by Shaker *et al.* [19]. The plates were dipped into petroleum ether, washed with ethanol, wiped with fine cloth, and allowed to dry in air. The dried glasses were numbered RC_1, RC_2, RC_3, RE_1, RE_2, RE_3, RN_1, RN_2 and RN_3. Where RC, RE and RN represent alkyd resins prepared from Clemson-spineless, Ex-samaru and NHAE-74 seed oils respectively. The numbers 1, 2 and 3 refer to the composition mixture of each alkyd resin containing 0.00, 0.50 and 1.0 g of maleic anhydride respectively.

2.4. Drying Time Test

To the 60 % resin solutions in xylene, a 1.0 g of drying catalysts [mixture of lead octanoate (0.41g) manganese octanoate (0.33 g) and cobalt octanoate (0.26g)] was added [20]. The glass plates were coated by submerging in the alkyd resin at room temperature until free from bubbles then suspended for drying in dust free atmosphere at room temperature [21].

The drying time of the alkyd resins were determined using Set-to-touch method described by [22] where a clean and dried finger was used to lightly touch the test film with the tip (the pressure of the fingertip against the coating shall not be greater than that required to transfer a spot of 3 to 5 mm of the coating to a cross section) and immediately placed the fingertip against a piece of clean, clear glass and observe if any of the coating was transferred to the glass. The film is set-to-touch when it neither adheres to the glass nor to the finger.

2.5 Abrasion Resistance Test

The Abrasion resistance test measures the resistance of a film against abrasion caused by an abrasive (silica sand) falling from a specified height through a guide tube onto a coated glass. The volume of sand which changes the thickness of the film by a certain extent determines the abrasion resistance. The abrasion is given in terms of amount of sand required to remove 1 µm thickness from coated material [12].

2.6 Adhesion Test

The dried films were cut by cross hatch then cleaned using soft brush to remove any detached flakes or ribbons of coating. Adhesive tape was placed on the cut area and rubbed firmly to ensure good contact. The tape was then pulled off rapidly back close to an angle of 180° as possible. The cross-cut area was inspected for the removal of coating from the substrate and rated as;0B (greater than 65 % area removed), 1B (35-65 % area removed), 2B (15-35 % area removed), 3B (5-15 % area removed), 4B (less than 5 % area removed) or 5B (0 % area removed) [23-24].

2.7 Chemical Resistance Test

The cured resin films were immersed in distilled water, 5 % $NaCl_{(aq)}$, 0.1 M $H_2SO_{4(aq)}$ and 0.1 M $NaOH_{(aq)}$ for 24 h at room temperature and weight lost was measured. An expression of the measurements; excellent (not affected), fair (not affected but change in colour), and poor (completely removed) was used to indicate the chemical resistivity of the film with respect to their weight loses [12-25].

2.8 Solubility Test

Approximately 0.5 g of each resin was measured and dissolved in 12.50 cm^3 of xylene, benzene, toluene, acetone, ethanol and methanol and allowed to stand for 5 min then filtered to remove suspended particle to obtain clear solution. The percentage solubility of the alkyd resin was estimated by quantitatively transferring the solution into a pre-weighed watch glass and evaporated the solvent at 100 ^oC then cooled in a desiccator containing silica gel [26]. The percentage solubility was calculated according to the equation 1:

$$\frac{\text{weight of Alkyd Resin after evaporation}}{\text{weight of the sample}} \times 100 \quad 2.1$$

2.9 Viscosity Measurement

The viscosity of the alkyd resins was measured by submerging a spindle (size 63) of the cannon viscometer instrument (model 2020) into 60 % solution of resin and resistance of the alkyd resin was measured at a speed of 20 rotations per minute at 31 ^OC (room temperature). The instrument calculates the result and directly displays the viscosity in centipoise (CP) [27].

2.10 Fourier Transform Infrared (FTIR) Spectroscopy

One microliter of diluted samples (1/100, v/v, in methylene chloride was injected manually in the slit less mode. The frequency and intensity of each band was obtained automatically by using the "find peak" command of the instrument software. The structural and functional group of okra seed oil-based alkyd resins was analyzed using Buck M530 Fourier Transform of Infrared Spectrophotometer (FTIR). The FT-IR equipment was operated in a frequency range of 4000–500 cm^{-1} [28].

2.11 Statistical Analysis

The results obtained from the analyses were reported as mean ± standard deviation of triplicate values. One way ANOVA was performed to identify significant differences among means at 5 % confidence level.

3. Results and Discussion

3.1 Physical Performance of Alkyd Resins

The results of the physical performances of the alkyd resins (set-to-touch drying time, abrasion resistance and adhesion tests) are presented on Table 3.1.

Table 3.1: Result of Physical Performance of Alkyd Resins synthesized from okra oil

Alkyd Resin	Drying Time (Min)	Abrasion Resistance	Adhesion Resistance
RC_1	82.50±0.71	NE	4B
RC_2	81.50±3.54	NE	4B
RC_3	82.50±2.12	NE	4B
RE_1	88.00±2.83	NE	4B
RE_2	90.00±8.49	NE	4B
RE_3	78.00±1.41	NE	4B
RN_1	85.00±1.41	NE	4B
RN_2	95.50±13.44	NE	4B
RN_3	83.00±0.00	NE	4B

*The result is expressed as mean ± standard deviation of triplicate result.

Where; RC = Alkyd resin from Clemson spineless;
RE = Alkyd resin from Ex-Samaru and
RN = Alkyd resin from NHAE-74
NE: No Visible Effect
4B: Less than 5% was removed

The synthesized alkyd resins have drying time in the range of 78.00 - 95.50 min, which is similar to the drying time (75 - 80 min) of alkyd resin from soya bean oil [25]. The obtained result is higher than that (6 – 48 min) reported for alkyd resin from rubber seed and linseed oil blend [30], but lower than (563 - 692 min) alkyd-based resin from non-drying oils [21]. The obtained drying time value of the resins is due the semi drying nature of the okra seed oil [30]; it shows inverse proportionality to the iodine value of each oil (higher the oil iodine value, lesser the time it requires to dry). An optimum drying time of 94.20 min is an important factor for the paint formulation [31-32]. If coating dries too fast, it will be prone to brittleness and if it dries too slowly, it is subjected to pick up dirt [33]. The drying time of all the alkyd resins analyzed gave satisfactory results [34]. Therefore, they can be used in paint formulation as a binder.

The abrasion resistance results of all the prepared alkyd resins showed no scratch on the alkyd resin by the falling sand at height of 1 m. The result is similar to the alkyd resin prepared from cotton seed oil [35]. The excellent resistance to abrasion may be attributed to the semi-drying nature of the okra seed oils used and level of the dryness of the resins. Timothy *et al.* [35] reported that the nature of the polybasic acids used may influence the abrasion resistance of alkyd resin.

The adhesion resistance test was to determine the strength of the bonds formed between the coating material and the applied surface. During adhesion resistance test, less than 5 % of each applied alkyd resin was removed. This showed similarity with the adhesion resistance of palm oil alkyd resins [23]; less resistance (0 % removed) compared to alkyd resins prepared from mixture of some oils [36] but more resistant than breadfruit seed oil modified alkyd resin [37]. The presence of –OH and –COOH (resulting from incomplete alcoholysis) functional groups can promote attraction between the substrate and alkyd resin during film formation due to their wetting and wetting maintenance properties of substrate surface [35,38]. The alkyd resins of in this study showed excellent adhesion property according to Nigerian Industrial Standards that recommend less than 50 % maximum removal of a dried film [37].

The result of chemical resistance test is presented in Table 3.2. The result showed that, distilled water and 5 % NaCl does not affect the resin completely. The 0.1 M H_2SO_4 changed the colour but does not remove the resins while 0.1 M NaOH completely dissolved the resins (i.e., completely washed away the resin from the material).

Table 3.2: Results of Chemical Resistance Test

Sample	Distilled Water	5% $NaCl_{(aq)}$	0.1 M $NaOH_{(aq)}$	0.1 M $H_2SO_{4(aq)}$
C.spineless	Excellent	Excellent	Poor	Fair
Ex-samaru	Excellent	Excellent	Poor	Fair
NHAE-74	Excellent	Excellent	Poor	Fair

Similar results were reported from breadfruit seed oil modified alkyd resins [37] and resin produced from shea butter oil [39]. However, Shaker *et al*. [19] and Islam *et al.* [21] reported the alkali resistance of modified alkyd resins from jojoba seed oil and alkyd-based resin from non-drying oil. The fair resistance of the resin to $H_2SO_{4\,(aq)}$ is due to reaching of equilibrium of the resin (ester) and its hydrolysed product (carboxylic acid and alcohol) while poor resistance against 0.1 M alkali solution may be due to the removal hydrolysed carboxylic acid by the reaction of $NaOH_{(aq)}$ (saponification reaction) which can reach completion [3540]. The resistance of water and 5 % NaCl solution is an indication of rigid network structural formation of the resin, which acts as barrier for penetrating water molecules through the surface of dried resin [41]. The water and acid resistance of a resin are very important factors for high coating durability during usage [42].

3.3 Solubility Test

The result of solubility tests is presented in Table 3.3. There is no significant difference ($p > 0.05$) in solubility of acetone among all the resins. The benzene solubility test showed that RN_2 is significantly more soluble ($p< 0.05$) than all the resins prepared, the solubility of RE_2, RC_2, RN_3, RC_3, RE_3 and RC_1 in benzene do not significantly differ ($p > 0.05$). The RC_2, significantly differ ($p < 0.05$) in solubility of ethanol with RE_1, and RN_1, but evidence of significant difference ($p > 0.05$) among the other resins. The solubility of resins in methanol and xylene showed no significant difference ($p > 0.05$) among themselves.

Table 3.3: Solubility Test (%)

	Solvents				
Alkyd Resin	**Acetone**	**Benzene**	**Ethanol**	**methanol**	**Xylene**
RC_1	0.99 ± 0.60^{a}	0.62 ± 0.01^{bcde}	1.02 ± 0.62^{ab}	0.15 ± 0.50^{a}	0.57 ± 0.43^{a}
RC_2	1.92 ± 0.24^{a}	1.16 ± 0.02^{bc}	2.47 ± 0.05^{a}	1.56 ± 0.08^{a}	0.53 ± 0.13^{a}
RC_3	1.38 ± 0.40^{a}	0.81 ± 0.16^{bcd}	1.83 ± 0.59^{ab}	0.65 ± 0.88^{a}	0.52 ± 0.30^{a}
RE_1	0.99 ± 0.01^{a}	0.03 ± 0.02^{e}	0.60 ± 0.30^{b}	0.71 ± 0.64^{a}	0.43 ± 0.16^{a}
RE_2	0.91 ± 0.12^{a}	1.17 ± 0.45^{b}	1.67 ± 0.57^{ab}	0.64 ± 0.48^{a}	0.45 ± 0.16^{a}
RE_3	0.76 ± 0.14^{a}	0.44 ± 0.10^{cde}	1.19 ± 0.04^{ab}	0.53 ± 0.04^{a}	0.58 ± 0.54^{a}
RN_1	1.28 ± 0.15^{a}	0.20 ± 0.08^{de}	0.65 ± 0.11^{b}	0.58 ± 0.16^{a}	0.89 ± 0.11^{a}
RN_2	1.62 ± 0.78^{a}	1.97 ± 0.09^{a}	1.14 ± 0.36^{ab}	0.38 ± 0.17^{a}	0.12 ± 0.01^{a}
RN_3	1.44 ± 0.33^{a}	1.04 ± 0.21^{bc}	1.57 ± 0.49^{ab}	0.77 ± 0.42^{a}	0.12 ± 0.01^{a}

*The result is expressed as mean ± standard deviation of triplicate result. Values in the same row that do not share a letter are significantly different ($p<0.05$)

The result revealed that alkyd resins prepared in this study are generally more soluble in protic solvent (acetone, methanol and ethanol) due to the presence of ester and hydroxide functional groups in the alkyd resins compared to the aprotic solvent. Solvents are added to overcome problem of high viscous resin which is an obstacle to substrates wet ability during application which may affect the bond between the alkyd resin and the substrate [41].

Fig. 3.1 shows the result of the viscosity measurement of the synthesized alkyd resin using oils of the three selected varieties of okra seed.

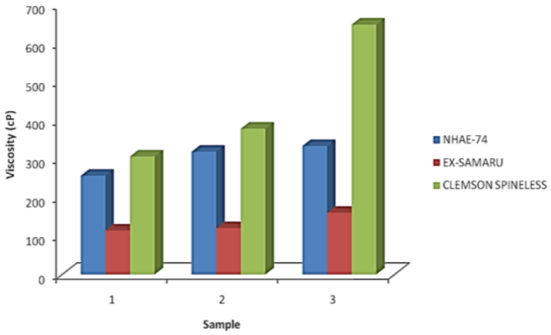

Figure 3.1: The result of viscosity measurements of Alkyd Resin

The result showed that, the viscosity of the resins produced are in the order using oil of NHAE variety is more viscos than that produced using oil of Ex-samaru but less viscos than the resin of Clemson spineless variety. The viscosity is proportional to the iodine value of the oil and quantity of maleic anhydride added. Viscosity increases with the number of cross-links, which means the formation of longer and branched polymer chains [23]. The maleic anhydride contains double bond in its structure; hence, there is a possibility of thermal polymerization with oil, which could bring about increased viscosity [44]. Low viscosity is important for controlling coating thickness and enabling proper wetting of the substrate [45]. Different suitable solvents are used for reducing the viscosity of high viscos resin. High viscos resin requires large volume of dilution solvent, thus reducing the cost of the alkyd [33].

3.4 Fourier Transform Infrared (FTIR) Spectroscopy

Major characteristic peaks in the IR spectra of the alkyd resins, along with their assignments, are shown in Table 4.10.

Table 3.4: Result for Fourier Transform Infrared (FTIR) Spectroscopy

Major Peaks (cm^{-1})	Assignment
3200-3550	$\text{O-H}_{\text{Str.}}$ (broad)
2500-3300	$\text{C-H}_{\text{str.}}$
1735-1750	$\text{C=O}_{\text{str.}}$ (ester)
1350-1470	$\text{C-H}_{\text{bend.}}$
735-770	$\text{C-H}_{\text{bend.}}$
1000-1300	$\text{C-H}_{\text{bend.}}$

Broad absorption peak at 3200-3550 cm^{-1} indicates the presence of hydroxyl group. The presences of C-H stretching and bending are identified at range of 2500-3300 cm^{-1} and 1350-1470cm^{-1} respectively. The formation of the ester bond is indicated by the presence of peak at 1,735–1,750 cm^{-1} for the C=O stretching. *Ortho*-di-substituted benzene shows a strong band in the region 735-770 cm^{-1} and aromatic C-H bending band is identified at the region of 1000-1300 cm^{-1} [46].

The infrared spectra of all the samples (appendices 1-9) are similar (appendices 1). Artists' alkyd paints and resins of Jordan valley tomato oil showed similar spectra [47,48]. Such spectrum serves as a finger print of the alkyd resin material [49]

4. Conclusion

Three different types of alkyd resins were prepared from Clemson spineless, Ex-samaru and NHAE-74 by varying composition of Maleic anhydride. Analysis of the alkyd resins synthesized showed the viscosity and FTIR result of obtained are comparable with alkyds obtained by many researchers and also commercial alkyds. Analysis of all the alkyd resins showed good performance characteristics, good resistance to distilled water and 5% sodium chloride solution, fair resistance to the attack of sulphuric acid, all the alkyds show more solubility in protic (acetone, methanol and ethanol) solvents than aprotic (benzene and xylene) solvents.

5. Recommendations

Further studies should be carried out for the best formulations of paints and varnishes with the produced alkyd resins with view of finding out the possibility of reducing the demands on the conventional oils in the paints industries.

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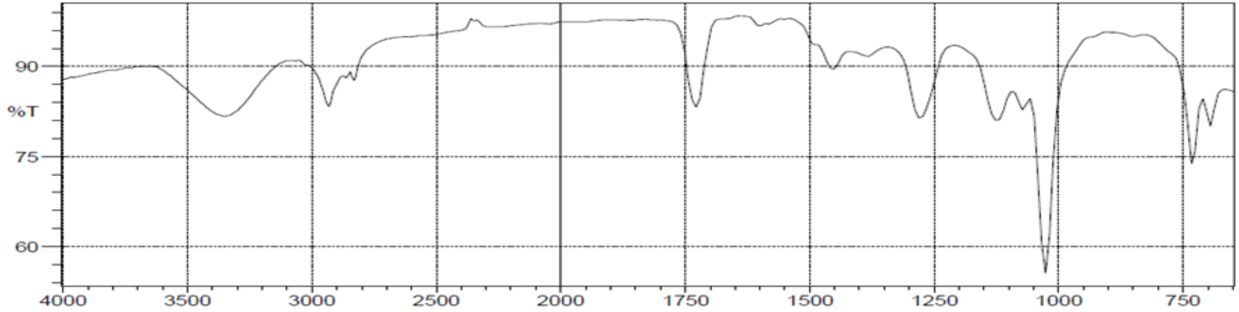
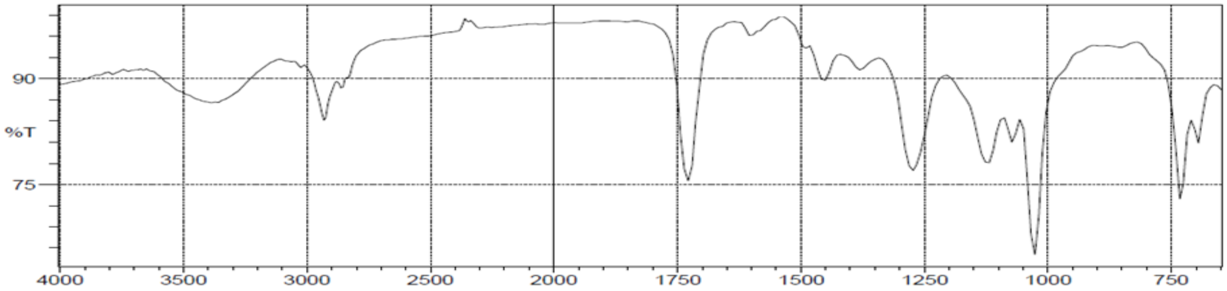
Appendices

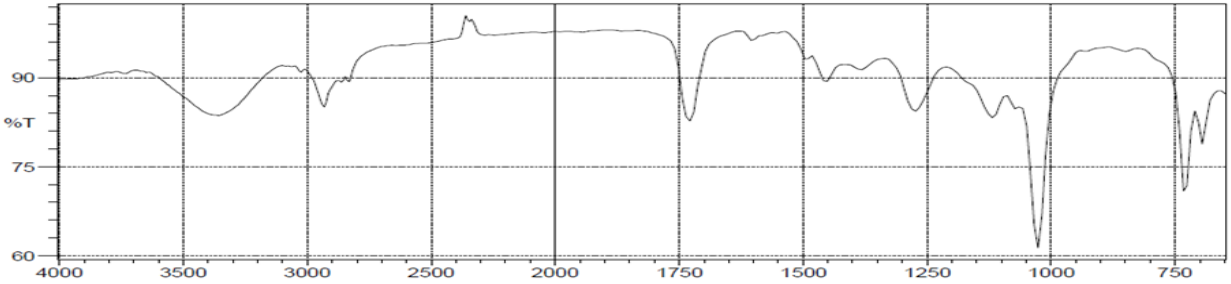
Appendix 1: FTIR Result of RC$_1$



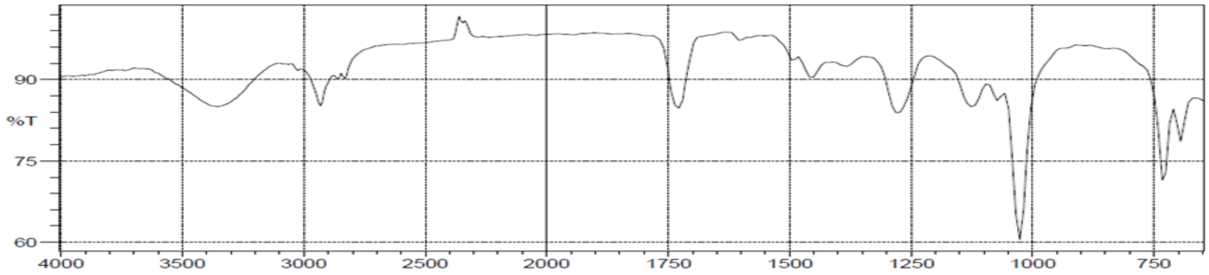
Appendix 2: FTIR Result of RC_2



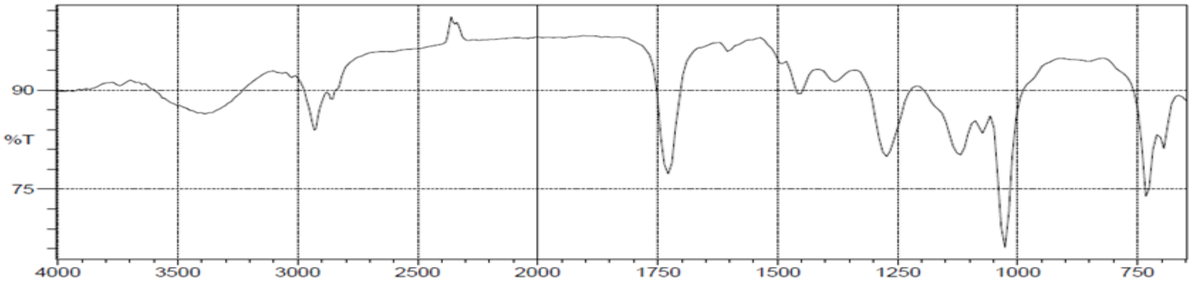
Appendix 3: FTIR Result of RC_3



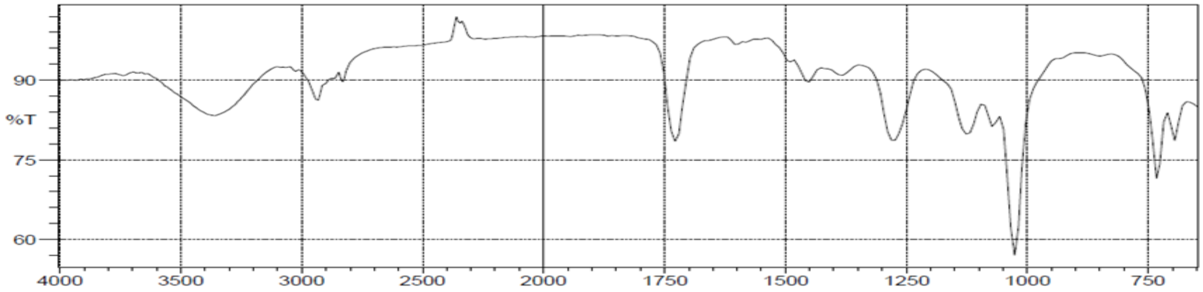
Appendix 4: FTIR Result of RE_1



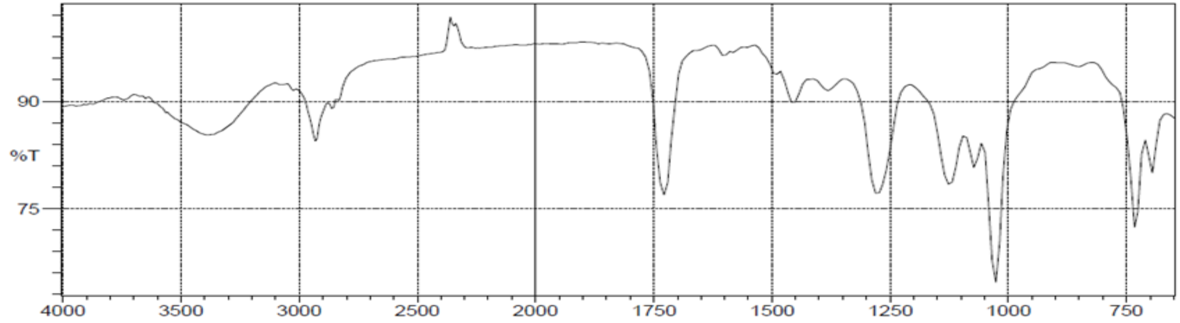
Appendix 5: FTIR Result of RE_2



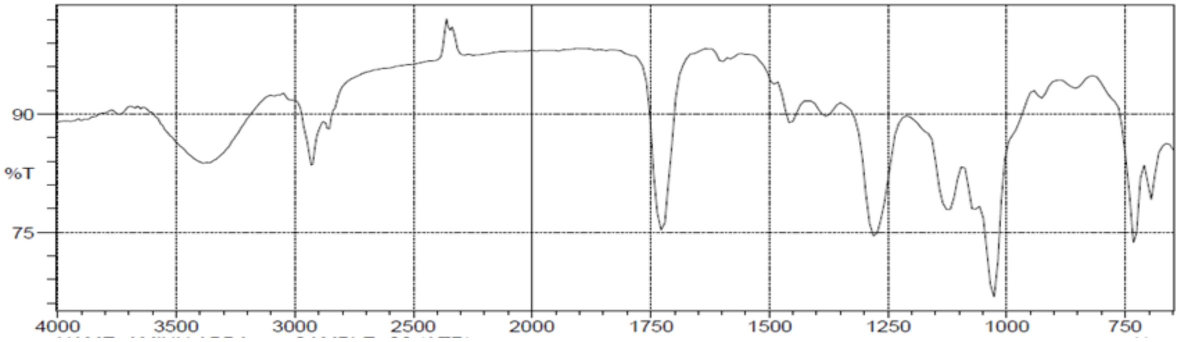
Appendix 6: FTIR Result of RE_3



Appendix 7: FTIR Result of RN_1



Appendix 8: FTIR Result of RN_2



Appendix 9: FTIR Result of RN_3