Current state of alkyd resin extraction, synthesis, and characterization

Abstract

Alkyd resins' high film-forming capacity, excellent adhesion, and durability to a variety of environmental conditions have led to its major attention in the field of industrial coatings and paint applications. The purpose of this work is to present a thorough overview of the synthesis and characterization of alkyd resins for these particular uses. Alkyd resins are made by reacting polyols (like glycerol or pentaerythritol) with dicarboxylic acids (like phthalic anhydride), then modifying the mixture with fatty acids or drying oils. The qualities of the resulting alkyd resins are significantly influenced by process variables, including catalysts, reaction conditions, and raw material selection. Numerous methods are used in the characterization of alkyd resins, such as nuclear magnetic resonance (NMR), Fourier transform infrared spectroscopy (FTIR), gel permeation chromatography (GPC), and thermal analysis (TGA/DSC).

These methods offer important insights into the alkyd resins' molecular weight distribution, thermal stability, and chemical structure. Gaining knowledge about the synthesis and characterization of alkyd resins helps to optimize their formulation for use in paint and industrial coatings, improving their performance and durability.

Keywords: Alkyd resin, Coating, Painting, Vegetable oils, TGA, DSC, FTIR, GPC, NMR

1 Introduction

Vegetable oils (VO) are extracted from oil containing seeds, nuts or fruits by various squeezing techniques, solvent extraction, or a blend of these; they are employed widely in modern applications like plasticizers, biodiesel, greases, glues, biodegradable bundling materials, printing inks, paints, and coatings. They are non-harmful, non-depletable, locally bountiful, stable, and biodegradable substances [1], [2]. Alkyd resins, a type of polyester derived from the condensation reaction between polyfunctional alcohols and polybasic acids, have gained significant importance in the formulation of industrial coatings and paint applications. The unique combination of properties, such as excellent adhesion, exceptional durability, high gloss, good weathering resistance, and compatibility with a variety of pigments and additives, make them a preferred choice in the coating industry [1], [3].The synthesis of alkyd resins involves a series of chemical reactions, including esterification, transesterification, and polycondensation, leading to the formation of polyester chains with various lengths and functionality. These reactions can be controlled and manipulated using different reaction parameters, such as reactant ratios, reaction time, temperature, and catalysts, to obtain alkyd resins with tailored properties [4], [5].These resins were used in the various fields of application like paint, coating, adhesives, and composite binder [1], [6], [7].

Various alkyd resins based on newly extracted oil having a high level of unsaturated acid had been constructed as a renewable raw material. Short (I), medium (II), and long (III) alkyd resins were made with oil, glycerol, and phthalic anhydride (PA) in varying proportions [1], [6]– [12].

Characterization of alkyd resins is essential not only to understand their structure but also to assess their suitability for different coating applications. Various techniques are employed to evaluate their molecular weight, functionality, chemical composition, thermal stability, mechanical properties, and film-forming behavior[1], [13].One of the important properties to evaluate during characterization is the molecular weight of the alkyd resin. This can be determined using techniques such as gel permeation chromatography (GPC) [14]or size exclusion chromatography (SEC) [15]. Molecular weight impacts the viscosity, film formation, and overall performance of the resin, making it a critical parameter to measure [1], [2], [14], [16].

Another important characteristic is the acid number, which indicates the amount of free fatty acids present in the alkyd resin. This parameter is crucial as high levels of free acids may lead to poor film formation and decrease the overall performance of the coating. Acid number can be determined by titration with an appropriate base, such as potassium hydroxide (KOH), and is expressed as the number of milligrams of KOH required to neutralize one gram of the resin [1], [10].Additionally, the drying time and film formation properties of the alkyd resin are vital for industrial coating applications [17]. These properties can be evaluated through techniques like the Gardner-Sward or König pendulum hardness tests, where the hardness and drying time of the resin film are measured. The adhesion strength of the alkyd resin to different substrates is also a critical consideration and can be assessed using techniques such as pull-off adhesion or cross-cut adhesion tests[14], [18].

It is crucial to synthesize alkyd resins with desired properties and understand their structure-function relationships to optimize their performance in industrial coatings and paint applications. This review aims to provide an overview of the synthesis methods and characterization techniques used for alkyd resins, highlighting their importance in achieving desired coating properties and performance.

2 Materials and method

Alkyd resin, vegetable oil, coating, paint, were employed as search terms in online search engines, these search engines included Google, Google Scholar, SciFinder, ScienceDirect, PubMed, Web of Science, Semantics Scholar and Researchgate. Thirty-eight (38) vegetable oils were reviewed from 2001 to 2023. Four (4) extraction methods (Soxhlet, mechanical press, distillation, and hot water floatation) were employed for the extraction of these vegetable oils.

3 Selection criteria for vegetable oils in alkyd resin synthesis

The selection criteria required for the suitability of the vegetable's oils in the synthesis of an alkyd resin are based on their inherent physicochemical properties such asiodine value, saponification value[1], [7], [9], [10], [13], [19].
Vegetable oils are divided into drying, semi-drying and non-drying based on their iodine values; which is expressed as grams of I_2 absorbed/100 g sample under standard conditions, iodine value is a measure of the unsaturation of oils and fats and their fatty acid derivatives and saponification values which depends on the molecular weight and the percentage concentration of fatty acid components present in the seed oil and it is effectively used to determine the average relative molecular mass of oilsand fats measure in in mg KOH/g[1], [7], [9]. Drying oils are expected to have their iodine values in the range 130 to 190 g I_2/100g which makes them able to dry rapidly whilst, semi drying oils are in the range of 100 to 130 g I_2/100g, below 130 g I_2/100g are considered non-drying oils and hence, not suitable for alkyd resin synthesis[1], [13], [17], [20]. Furthermore, it has been suggested that high saponification values imply that the oil possess small amounts of impurities [21].
The vegetable oils investigated in this study are linseed oil [22]–[26], castor seed oil[27]–[30], soybean seed oil [27], [30]–[34], sunflower seed oil [35], [36],tung seed oil[37], rubber seed oil [38]–[40], Jatropha curcas linneaus seed oil [41]–[47],tomato seed oil [48], wild olive seed oil [17], Albiza lebbeck seed oil [49], benniseed oil [50], breadfruit seed oil [51], palm kernel seed oil [52]–[56], Hura crepitans seed oil [55], [57], Luffa cylindrica seed oil [58], African walnut seed oil)[59], camelina sativa seed oil [25], locust-bean seed oil [38], Parinari-polyandra benth seed oil [60], LagenariaBreviflorusseed oil [61], yellow oleander seed oil [62], Cotton seed oil [55], [63], [64],Delonix Regia and Theventia Peruviana Seed Oils [65], [66], Luffa aegyptiaca seed oil[67], Jojoba seed oil [68], *Citrullus colocynthis* seed Oil[69], Nahar seed oil(*Mesua ferrea*)[70], [71], Sesame seed oil [4], [65], sweet almond seed oil [44], *Acacia auriculiformis* seed oil[72]. Karanja (Millettia pinnata (l.) panigrahi) seed oil [42], plukenetia volubilis seed oil [26], *Picralima nitida* seed oil [73],Rapeseed oil [47], Gmelina seed oil [74], ricinodendron heudelotii oil[75], Sancha inchi (Plukentia volubilis L.) [76], Okra seed oil [77].
It was recorded that Sancha inchi vegetable oil had the highest iodine value of 189.16g I_2/100g[76]which was followed by the linseed oil, with an iodine value recorded at 177.00 g I_2/100g and 188.00 g I_2/100g[23], [40]. The least iodine values were found to be 22.10 g I_2/100gand 34.90 g I_2/100g for cotton seed and Gmelina seed oil [64], [74]. It has been suggested that oils with higher proportions of polyunsaturated fatty acids (PUFAs), such as linoleic acid (C18:3 and C18:2), are likely to have higher iodine values[44], [72]. Examples of such vegetable oils composed of high iodine values (> 120 g I_2/100g) recorded in this investigation enumerated in Table 1 are Luffa aegyptiaca [67], Jatropha curcas linneaus seed oil [45], Sesamum indicum seed oil [4], Acacia auriculiformis seed oil [72], Soybean seed oil [30], [31], [72], Ricinodendron heudelotii [75], Hura crepitans (sandbox) [55], [57], Delonix regia (Flamboyant) [66], Citrullus colocynthis [69], Cotton seed oil [55], Rubber seed oil [38]–[40], Jordan valley Tomato [48], African walnut seed oil [59], Ximenia americana [5] and Luffa cylindrica oil [58]as they are composed of reasonable amount of PUFAs [64]. On the other hand, vegetable oils with a higher composition of saturated fatty acids (SFAs), such as investigated in this study;Locust-bean [38], Jatropha curcas linneaus [43], [78], Yellow

oleander (Thevetia peruviana)[62],[86]Castor seed [27]–[30], Mangifer Indica [79], Karanja (Millettia pinnata (L.) Panigrahi) [62], Breadfruit seed [51], Gmelina seed [74], Nahar seed (Mesua ferrea) [70], [71], *Lagenaria Breviflorus* seed [58], Palm oil [52], Jojoba seed [68], Neem (*Azadirachta indica*) oil [80], tend to possess low iodine values (< 120 g I_2/100g)[81].Whilst the saponification values ranged from 38.23 mg KOH/g in Gmelina seed oil [74]to 241 mg KOH/g in Nahar seed (Mesua ferrea) oil [71]. It has also been suggested that oils with high levels of unsaturated fatty acids, such as linoleic acid and oleic acid, tend to have higher saponification values. This is because unsaturated fatty acids have more reactive sites (double bonds) that can be easily hydrolyzed by alkali, resulting in a higher saponification value. On the other hand, oils with high levels of saturated fatty acids, such as stearic acid and palmitic acid, have lower saponification values[64].

The molecular weight of the fatty acids present in vegetable seed oils also influences their saponification values. Oils with higher molecular weight fatty acids generally have higher saponification values[64]. This can be attributed to the higher molecular weight fatty acids requiring more of the alkali to break and saponifytheir ester bonds.

4 Extraction methods of vegetable oils

The various extraction techniques employed in this study as seen in Table 1 encompassed; Soxhlet extraction [27], [29]–[31], [33], [39], [42], [44], [46], [49], [51], [55], [60], [62], [65], [66], [69], [72]–[74], [77], [79], hot water floatation [4], [82],Mechanical press [47], [50], distillation [58] whilst, [22], [26], [28], [32], [34], [35], [43], [64], [76], [83]had their vegetable oils purchased.

It was discovered that 61.5% of Researchers employed the Soxhlet extraction technique, 28% of Researchers purchased their vegetable oils, 5.3% of Researchers employed the mechanical expression method, 2.6% of Researchers employed the hot water floatation and distillation methods respectively. The Soxhlet extraction method was widely employed for the extractions of *Luffa aegyptica* seed oil [67], Albizia benth seed oil [49], Castor seed oil [27], [29], [30], Jatropha curcas Linneaus seed oil [42], [46], [65], Sesame seed oil [65], [82], sweet almond [44], *Acacia auriculiformis* seed oil[72], Soybean seed oil [27], [30], [31], [33], [72], *Picralima nitida* seed oil [73],Delonix Regia seed Oils [66], *Citrullus colocynthis* Seed Oil[69], Cotton seed oil [55], *Hura Crepitans* seed oil [55], Palm kernel seed oil [55], Mango seed oil [79], Rubber seed oil [39], Tomato seed oil [48], Karanja (Millettia pinnata (L.) Panigrahi) seed oil [62], Parinari-polyandra benth seed oil [60], Breadfruit Seed Oil[51], Neem (*Azadirachta indica*)[80], Okra seed oil [77]and Gmelina Seed Oil [74]respectively. Hot water floatation method was adopted by [4], [82]for sesame seed oil, Mechanical press method was adopted by [47], [50]for Beniseed oil, Jatropha curcas Linneaus seed oil and Rapeseed oil respectively, on the other hand, distillation method of extraction was employed by [61]for Lagenaria*Breviflorus* seed oil.

Based on the vegetable oils studied, 23 researchers employed the use of Soxhlet for their extractions [27], [29]–[31], [33], [39], [42], [44], [46], [49], [51], [55], [60], [62], [65], [66], [69], [72]–[74], [77], [79].

Mechanical press extraction technique was next to Soxhlet extraction based on the frequency as reported in the literature. It was observed that 2 Researchers reported using this technique [47], [50]. Only 1 Researcher employed the distillation method and hot water floatation methods for their extraction [61], [82] whereas, 10 Researchers had their vegetable oils purchased [22], [26], [28], [32], [34], [35], [43], [64], [76], [83]

The wide use of Soxhlet extraction method could be attributed to its notable high extraction efficiency especially for extracting oil from solid materials such as seeds or plant materials as it allows for thorough and complete extraction of oil, resulting in a higher oil yield compared to other methods. It also allows for selective extraction of specific compounds, including oils, from the solid materials [27], [33], [72]. It provides the ability to target and extract specific components of interest, which can be advantageous in certain applications. It also utilizes a continuous extraction process with a solvent, typically an organic solvent like hexane or ethanol [44], [72]. One of the advantages of this method is that it allows for the recycling of the solvent. The solvent vaporizes, condenses, and re-circulates through the extraction chamber, minimizing solvent waste and reducing costs[48]. The Soxhlet extraction process can be automated, allowing for continuous extraction with minimal human intervention. This saves time and labor compared to manual extraction methods and finally, the Soxhlet method operates at relatively low temperatures and is suitable for extracting heat-sensitive compounds[51], [60]. This is beneficial when extracting oils or compounds that may be affected by high temperatures used in other extraction processes like distillation[78]. It is worthy to note that when compared to mechanical press, hot water floatation and distillation methods, the choice of extraction method depends on various factors such as the nature of the raw material, target compounds, scale of production, equipment availability, and economic considerations.

5 Formulation of alkyd resin

The general formulation method for a suitable vegetable oil were outlined by[17], [82], [84]. Parameters such as functionality, molecular weight, equivalent weight, equivalent mole, number of acid equivalent, number of moles and amounts of reagents charged were considered.

5.1 Functionality (F)

This is the number of functional groups present in the seed oil, vegetable oils are triglycerides (esters), and are therefore said to have a functionality of 1, glycerolhave a functionality of 3 whilst, pthalic anhydride is said to have a functionality of 2[4], [17], [82], [84]

5.2 Molecular weight (M_w)

The molecularweights of the seed oil, polybasic acid, such as phthalic anhydride, and a polybasic alcohol (such as glycerol) are calculated, equation enumerates how to calculate the molecular weight of the seed oil whilst the molecular weights of the phthalic anhydride and polybasic alcohol are derived from their individual atoms sum[4], [17], [82].

$$\text{Molecular weight of seed oil} = \frac{56.11 \times 10^3}{\text{Saponification value}} \qquad (1)$$

Sebacic, isophthalic, and fumaric acids are further polybasic acids[4], [17], [82].

5.3 Equivalent weight (E_w)

The equivalent weights of the phthalic anhydride and polybasic alcohol are derived by dividing their molecular weights by their functionality whilst, the equivalent weight of the seed oil is equivalent to its molecular weight [17], [82]

5.4 Equivalent mole (E_m)

The equivalent moles of the seed oil, phthalic anhydride and polybasic alcohol is the derived by dividing the required weight in grams by their equivalent weights[4], [17], [40], [82].

5.5 Number of acid equivalent (N_a)

The number of acid equivalents is a measure of the equivalent mole of the acidic chemical species only, usually, the seed oil and pthalic anhydride[17], [27], [82].

5.6 Number of moles (N_0)

This is derived by dividing the measured weights of the seed oil, phthalic anhydride and polybasic alcohol required for the formulation by their individual molecular weights[17], [40], [82].These parameters essentially account for the weight-average molecular weight of the alkyd resin to be synthesized.The average molecular weight (M_{av}) with respect to the acid value at different stages of the synthesis can be calculated using equation 2:

$$\text{Mav} = \frac{W}{(\text{k} - \text{P})\,\ell\text{A}} \qquad (2)$$

where,W is the total weight of all the reactants charged, k is the ratio of the total moles of all reactants to total equivalent of the acid (N_o/N_a), ℓA is the total equivalent of the acid and P is the extent of reaction

The extent or fraction of reaction is defined as the fraction of the hydroxyl or carboxyl functional group that has taken part in the reaction at a given time.

The extent of reaction, *P*, with respect to the acid value can be calculated using the equation:

$$p = \frac{C_0 - C_t}{C_0} \tag{3}$$

C_0 is the acid value at zero time (t=0), C_t is the acid value at attained time (t=t).

Whilst, the average degree of polymerization, D_P, is calculated using the equation:

$$Dp = \frac{1}{1 - p} \tag{4}$$

[4], [17], [52], [82].

6 Synthesis methods of alkyd resins

It has been reported that over 90% of the literature reviewed in this work employed the alcoholysis polyesterification process of the synthesis of alkyd resins. This method was carried in two distinct stages:

a) Alcoholysis stage

This is the first stage in alkyd resin synthesis which is followed by the transesterification process [17], [31], [33], [74], [82]. This stage converts a calculated amount of the vegetable oil (a triglyceride) to a monoglyceride on reacting with a polyol, usually, glycerol with the aid of aninorganic catalyst usually CaO. The process is being monitored by taking aliquots at intervals to determine for the solubility in 1:3 volumes of methanol raised gradually to an elevated temperature of 200 ℃ and maintained for 2 hours[4], [33], [38], [82]. When this is achieved, a monoglyceride is thus formed and the system is allowed to cool to about 100 to 120 ℃. It is also essential to noted that the process is carried out in an inert system to prevent any oxidation reactions which eliminates oxygen from the reaction mixture by preventing the degradation of the alkyd resin and to control the reaction environment efficiently[17], [27], [82].
The schematic representations of alcoholysis of polyol (glycerol) andmonoglyceridepolyesterification of vegetable oil sample is given in Figures 1 and 2 [82].

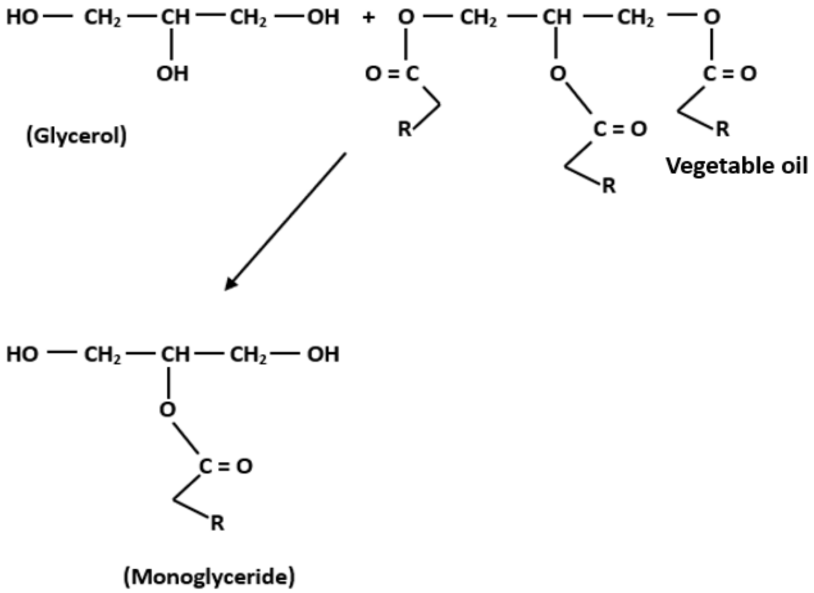

Figure 1 Alcoholysis of polyol and vegetable oil

b) Poly-esterification

This stage is a polymerization process that involves the removal of water, it can be termed a 'polycondensation process' or a 'transesterification process' as depicted in Figure 2.

This stage was initiated by the addition of a dibasic acid usually, phthalic anhydride to the reaction mixture, followed by addition of xylene which represents 10 % of the total alkyd resin weight to aid eliminate the water of esterification by producing an azeotrope [17], [39], [50], [82]. The temperature was extended to 230-250 ºC while the reaction progressed. Aliquots were taken from the reaction blend at time periods of 30 minutes in other to determine the drop-in acid value and volume of water removed were also recorded. The drop-in acid values were determined by titrating two aliquot portions of the mixture withdrawn against 0.1M KOH solution, a measured amount of phenolphthalein dissolved in a mixture of ethanol and toluene (1:1) [17], [82]serves as indicator. The reaction was halted when the acid value reached the value of less than 10 mg KOH g^{-1}[82].

The presence of drying oil in the reaction mixture helps to introduce unsaturation into the resin. This unsaturation allows for cross-linking and improved film-forming properties of the alkyd resin [17], [38], [82]. The polycondensation reaction involves the reaction of the ester groups, resulting in the formation of long polymer chains. This step is typically carried out at higher temperatures (230 °C to 250 °C) under reduced pressure to facilitate the removal of volatile byproducts[17], [27], [82]. It is worth noting that the choice of raw materials, catalysts, and reaction conditions can vary depending on the desired properties of the alkyd resin. Different combinations of polyols, polyacids, and drying oils can be used to achieve specific characteristics such as hardness, flexibility, or gloss[4], [27], [54], [82].

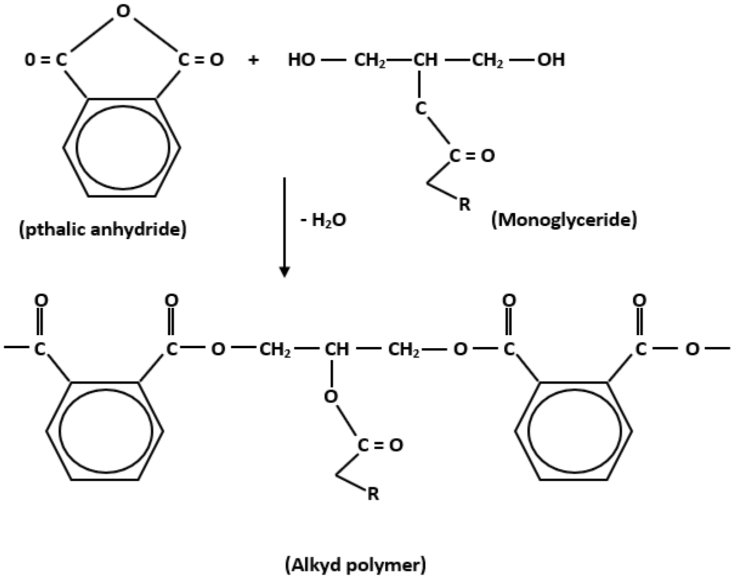

Figure 2 Polyesterification of monoglyceride

The fatty acid process was also reported by [26], [76]for the synthesis of alkyd resin from Sancha inchi (*Plukentia volubilis L.*) seed oil. Fatty acid fractions were obtained from oils using a base-catalyzed transesterification reaction[76].

The fatty acid process of alkyd resin synthesis involves the reaction of fatty acids with a polyhydric alcohol, such as glycerol or pentaerythritol, in the presence of an organic acid catalyst[76], [83]. This process is commonly used in the production of long oil alkyd resins.The synthesis begins with the selection and preparation of the raw materials. Fatty acids, such as linoleic acid or oleic acid, are derived from natural oils like linseed oil or soybean oil. These oils are subjected to a process called saponification, where the oil is heated with an alkali, such as sodium hydroxide, to break it down into its constituent fatty acids. The resulting fatty acids are then purified and separated[58], [76], [83].

In the next step, the purified fatty acids are heated with a polyhydric alcohol, typically glycerol or pentaerythritol[4], [17], [27], [82]. The reaction is typically carried out at a high temperature (around 200-220°C) in the presence of an organic acid catalyst, such as toluene sulfonic acid or para-toluenesulfonic acid[17], [76], [82]. The catalyst helps to speed up the reaction and improve the yield of the alkyd resin[17], [27], [65], [82].

During the reaction, the fatty acids undergo esterification with the polyhydric alcohol, resulting in the formation of ester bonds between the fatty acid chains and the alcohol[5], [65], [82]. This esterification reaction leads to the formation of a polyester backbone, giving the alkyd resin its structure.

According to [64], the alcoholysis polyesterification process offers better control over the resin properties compared to the fatty acid process. The researchers mentioned that by selecting appropriate alcohols and acids, it is possible to easily modify the resin's molecular weight, hydroxyl value, and acid value.

It has been suggested that the alcoholysis polyesterification process allows the use of various types of alcohols, including polyols, which can result in alkyd resins with improved properties such as higher flexibility and better water resistance[17], [39], [52], [82]. The investigation carried out by [37]found that alcohols used in alcoholysis provided faster reaction rates compared to fatty acids, resulting in shorter reaction times and higher production rates.

Another advantage of the alcoholysis polyesterification process is its potential for using renewable feedstocks. It was enumerated that this process allows the utilization of biomass-based alcohols, which can contribute to the sustainable production of alkyd resins[64]. Itcan be concluded that the alcoholysis polyesterification process of alkyd resin synthesis is preferable to the fatty acid process due to its better control over resin properties, versatility in choosing alcohols, faster reaction kinetics, and the potential for using renewable feedstocks.

Table 1 Exploring Vegetable Oils: Iodine Value, Saponification Value, Extraction Techniques and Synthesis Methods

S/n	**Seed oil**	**Wijs Iodine value (g I_2/100g) [VO]**	**Wijs Iodine value (g I_2/100g) [AR]**	**Saponification value (mg KOH/g) [VO]**	**Saponification value (mg KOH/g) [AR]**	**Method of extraction**	**Method of synthesis**	**Reference**
1	Luffa aegyptiaca	123.28	NA	178.50	NA	Solvent extraction using n-hexane	Alcoholysis and poly-esterification	[67]
2	Locust-bean	104	NA	176.00	NA	Solvent extraction using petroleum spirit	Alcoholysis and poly-esterification	[38]
3	Albizia benth	NA	NA	NA	NA	Solvent extraction using n-hexane	Alcoholysis and poly-esterification	[49]
4	Sweet almond	NA	70.96 ± 3.89	NA	246.845 ± 3.91	Solvent extraction using n-hexane	Alcoholysis and poly-esterification	[44]
5	Jatropha curcas linneaus	NA; NA; NA; 130.00; NA; 105.00; 62.40	65.015 ± 4.41; NA; NA; NA; NA; NA; NA	NA; NA; NA; 178.00; NA; NA; 196.50	256.29 ± 5.38; 148.42±0.27; 383.00; NA; NA; NA; NA	Solvent extraction using n-hexane/petroleum ether * Purchased	Alcoholysis and poly-esterification	[43]–[46], [65], [78]*[77]
6	Sesamum indicum	NA, 122.33; NA	84.065 ± 1.05; 74,83; NA	226.876 ± 3.45; 172.13; NA	282.45 ± 2.65; 246.80; 182.55±0.2	Solvent extraction using n-hexane; Hot water floatation	Alcoholysis and poly-esterification	[4], [44], [65], [82]
7	Yellow oleander (Thevetia peruviana)	71.4, 12.6	36.98, NA	121. 41.23	NA, NA	Solvent extraction using petroleum ether	Alcoholysis and poly-esterification	[42], [86]
8	Acacia auriculiformis	128.10	NA	193.80	NA	Solvent extraction using n-hexane;	Alcoholysis and poly-esterification	[72]
9	Soybean	126.6; 127.9; NA; 128.40; 131.00 ± 0.11; NA; NA; NA; NA; NA;	NA; 28.46; NA; 35.82; NA; NA; NA; NA; NA; NA; NA	193.28; 195.00; NA; 189.20; 192.00 ± 1.12; NA; NA; NA; NA; NA; NA	NA; 638.41; NA; 228.608; NA; NA; NA; NA; NA; NA; NA	Solvent extraction using n-hexane/ Purchased	Alcoholysis and poly-esterification	[27], [30]–[34], [72], [85]

		NA						
10	Ricinodendron heudelotii	156.82	NA	189.24	269.32	Solvent extraction using n-hexane;	Alcoholysis and poly-esterification	[75]
11	Linseed (Linum usitatissimum L.).	177.00; 188.00; NA; NA	NA; NA; NA; NA	204.00; 194.00; NA; NA	NA; NA; NA; NA	Solvent extraction using n-hexane/ Purchased	Alcoholysis and poly-esterification	[22]–[24], [40]
12	Castor seed	82.50; 82.80; 89.01 ± 0.21; 87.60	24.65; 36.33; NA; NA	189.00; 182.00; 181.50 ± 0.18; 169.34	636.42; 369.35; NA; NA	Solvent extraction using n-hexane/Purchased	Alcoholysis and poly-esterification	[27]–[29]
13	Hura crepitans (sandbox)	122.08 145.90	NA; 85.02	210.38 210.30	NA; NA	Solvent extraction using n-hexane; petroleum ether	Alcoholysis and poly-esterification	[55], [57]
14	Picralima nitida	NA	NA	NA	NA	Solvent extraction using n-hexane	Alcoholysis and poly-esterification	[73]
15	Delonix regia (Flamboyant)	139.62	48.84	230.15	302.94	Solvent extraction using n-hexane	Alcoholysis and poly-esterification	[66]
16	Citrullus colocynthis	128.26	NA	192.35	NA	Solvent extraction using n-hexane	Alcoholysis and poly-esterification	[69]
17	Cotton seed	125.13; 22.10; NA	NA; 43.86; 35.73	188.23; 63.19; NA	NA; 372.00; 357.64	Solvent extraction using petroleum ether / Purchased	Alcoholysis and poly-esterification	[55], [63][64]
18	Mangifer Indica (Mango)	50.91	NA	218.38	333.00	Solvent extraction using petroleum ether	Alcoholysis and poly-esterification	[79]
19	Rubber seed	136.20; NA; 142.20; 135.36	65.64; 67.23; NA; NA	181.14; NA; 180.40; 206.20	345.10; 173.40; NA; NA	Solvent extraction using n-hexane	Alcoholysis and poly-esterification	[39], [40], [84]
20	Jordan valley Tomato	123.00	NA	192.00	NA	Solvent extraction using petroleum ether	Alcoholysis and poly-esterification	[48]
21	African walnut (Tetracarpidium conophorum)	153.05	84.02	203.45	320.02	Solvent extraction using n-hexane	Alcoholysis and poly-esterification	[59]
22	Karanja (Millettia pinnata (L.) Panigrahi)	89.9	42.86	182	NA	Solvent extraction using petroleum ether	Alcoholysis and poly-esterification	[62]
23	Parinari-polyandra benth	80.71	NA	118.20	NA	Solvent extraction using n-hexane	Alcoholysis and poly-esterification	[60]
24	Ximenia americana (Wild Olive)	152.28	70.06 +0.74	178.5	297.5±1.48	Solvent extraction using petroleum	Alcoholysis and poly-esterification	[17]

						ether		
25	Breadfruit seed	107.30	NA	161.30	NA	Solvent extraction using n-hexane	Alcoholysis and poly-esterification	[51]
26	Gmelina seed	34.90	125.5	38.23	143.61	Solvent extraction using n-hexane	Alcoholysis and poly-esterification	[74]
27	Nahar seed (Mesua ferrea)	NA; 89.26	59.72.00; 93.00	NA; 241.00	263.00; NA	Distillation using petroleum ether; Hydraulic pressing	Alcoholysis and poly-esterification	[70], [71]
28	Beniseed	120.00	NA	NA	NA	Hydraulic pressing	Alcoholysis and poly-esterification	[50]
29	Rapeseed	NA	NA	NA	NA	Hydraulic pressing	Alcoholysis and poly-esterification	[47]
30	Luffa cylindrica	153.00	NA	202.00	NA	Distillation using n-hexane	Alcoholysis and poly-esterification	[58]
31	*Lagenaria Breviflorus* seed	110.0	NA	213.18	NA	Distillation using n-hexane	Alcoholysis and poly-esterification	[61]
32	Sancha inchi (Plukentia volubilis L.)	NA; 189.16	NA; NA	NA; 189.60	NA; NA	Purchased	Fatty acid process; Alcoholysis and poly-esterification	[26], [76]
33	Palm oil	NA; NA; NA; 45	NA; NA; NA; NA	NA; NA; NA; NA	NA; NA; NA; NA	Purchased	Alcoholysis and poly-esterification	[52]–[54], [56]
34	Sunflower	118.91 ± 0.40; NA	NA; 164.00	191.87 ± 0.60; NA	334; 190	Purchased	Alcoholysis and poly-esterification	[35], [36], [68]
35	Jojoba seed	95.00	NA	100.00	NA	Purchased	Alcoholysis and poly-esterification	[68]
36	Tung oil	NA	NA	192	NA	Purchased	Alcoholysis and poly-esterification	[37]
37	Neem (*Azadirachta indica*)	76.14	NA	176.40	NA	Solvent extraction using petroleum ether	Alcoholysis and poly-esterification	[80]
38	Okra seed	NA	NA	NA	NA	Solvent extraction using petroleum ether	Alcoholysis and poly-esterification	[77]

***NA =** Not available

2 Instrumental characterization techniques of alkyd resin

The characterization methods identified in this investigation include Fourier-transform infrared (FT-IR) spectroscopy, nuclear magnetic resonance (NMR), gel permeation chromatography (GPC), differential scanning calorimetry (DSC), Scanning electron microscopy (SEM) and thermogravimetric analysis (TGA).

FT-IR was the most used characterization technique in this study and this can be attributed to its availability and low-cost of usage and ease of interpretation which focuses on the determination of functional groups inherent in the vegetable oil/alkyd resin [28], [30], [34], [35], [42]–[44], [47]–[49], [61], [65], [66], [69], [74], [77], [79], [82].
Nuclear magnetic resonance which studies the structure, composition and dynamics of the alkyd resin molecules was adopted for the characterization of the vegetable oils/alkyd resin by the following researchers [22], [35], [46], [48], [49], [62], [69], [76]. It was discovered that only four researchers employed the differential scanning calorimetrywhich helped to ascertain the alkyd resins phase transitions such as melting, glass, crystallization and solid-state transformations in their molecules[4], [34], [48], [82]and thermogravimetric analysis was employed to determine their thermal stabilities and evaluation of their oxidations and decomposition reactions[41], [42], [82] each for their synthesized alkyd resin whilst, gel permeation chromatography was adopted by[76]in order to ascertain for alkyd resin molecular weight and a researcher employed the scanning electron microscopy to study the morphology of the synthesized alkyd resin [82].

Conclusion

In this study, 74 data were enumerated from 2001 to 2023 on the synthesis of alkyd resin from vegetable oils.This review was made to shed light on theirextraction methods, synthesis, and methods of characterization of alkyd resins formulated from these alkyd resins.The suitability of extracted vegetable oils is based on their inherent iodine and saponification values. This goes far as to ascertaining the degree of polymerization of these vegetable oils with suitable alkyd reagents.It is worthy to note that limited information based on the iodine values of the extracted vegetable seed oils were evident by the investigated Researchers, as such, the suitability of these vegetable oils could not be fully ascertained for some species of the extracted seed oils. 29 data collected revealed that the various species of vegetable seed oils extracted for alkyd resin synthesis had no iodine values investigated, nonetheless, 45 Researchers took their iodine values into consideration. It was also discovered that 44 Researchers enumerated the saponification values of their extracted vegetable seed oils whilst, 30 of these Researchers did not offer information on their vegetable seed oils.In this investigation it can be established that the extraction methods employed for the vegetable seed oils follow this trend;Soxhlet extraction >mechanical press > hot water floatation and distillation methods whilst, 11 Researchers had their vegetable oils purchased for the alkyd resin synthesis.
Result from this investigation found that only 2 Researchers employed the Fatty acid process of alkyd synthesis out of 74 data studied which suggests that the alcoholysis-polyesterification processoffer better control over resin properties, versatility in choosing alcohols, faster reaction kinetics, and the potential for using renewable feedstocks.The various characterization techniques adopted in this study revealed that the commonly used instrumentation was the FT-IR which was followed by the use of an NMR whilst, DSC, TGA, GPC and SEM were less commonly used for their alkyd resin characterization.

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