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Extraction, Synthesis, Characterization and Anti-Bacterial Studies of Garlic Extract-Modified Nanoparticles in Alkyd Resin Derived from Castor and Walnut Seed Oils

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ABSTRACT

This study explored the synthesis of alkyd resin from castor and walnut seed oils modified with CuO, ZnO and TiO_2 nanoparticles, as well as garlic extract to determine the synergistic antibacterial potency of the synthesized resin. The alkyd resin synthesized, using alcoholysis and esterification reactions, was evaluated via standard analytical methods including Gas Chromatography-Mass Spectrometry (GCMS), Association of Analytical Chemists (AOAC) methods, and the Fourier Transform Infrared (FT-IR) spectroscopy. The castor-alkyd resins exhibited a final acid value of 8.84 mg/KOH and a functional group conversion rate of 77% P, indicative of efficient polymerization and excellent film-forming capabilities. The walnut alkyd resin, showed the lowest acid value (8.18 mg/KOH) and a comparable conversion of 76%. The incorporation of metal oxide nanoparticles modified with garlic extract of CuO/ZnO/TiO_2, significantly enhanced the antimicrobial properties of the alkyd resins. The castor alkyd resin, modified with garlic extract nanoparticles, exhibited the highest antibacterial efficacy resulting in inhibition zones as large as 49.5 mm. The walnut oil-based alkyd resin, demonstrated notable antibacterial activity against Escherichia *coli* and Staphylococcus *aureus*, especially when modified with ZnO/TiO_2 and CuO/ZnO nanocomposites. This synthesis have proven to be cost-effective and a renewable modified feedstock for alkyd resin suitable for antimicrobial coating applications.

Keywords: seed oils, alkyd resin, coating, nanoparticles, anti-bacterial, garlic extract.

INTRODUCTION

The transmission of infectious agents via contaminated surfaces is a critical problem in public spaces and environments, with significantly high risk of spreading microbes, such as bacterial and viruses [1]. Surfaces contaminated by these microbes play a vital role in the indirect transmission of infections. Antimicrobial coatings have been developed and designed using

petroleum-based synthetic polymers and incorporated with metal oxide nanoparticles such as TiO_2, ZnO, CuO [2], to enhance antibacterial properties of the coatings, which prolonged activity by disrupting and inhibiting bacterial membranes in order to reduce bacterial viability [3, 4]. The overuse of petroleum-based coatings has raised serious environmental concerns as their CO_2 emission depletes natural resources and causes pollution including oil spills [2, 5]. To address these sustainability issues, researchers are exploring bio-based alternatives for polymer synthesis [6].

The advent of green chemistry technology enabled the development of bio-based coatings with properties similar to petroleum-based counterparts, in producing alkyd resins [7]. Researchers have used vegetable oils such as castor oil and walnut oil, as renewable feedstock in the coating industry [8]. Vegetable-based oils, derived from castor oil, have demonstrated excellent hydroxyl content for effective crosslinking, ensuring strong mechanical and chemical performance.

Walnut oils derived from *Juglans regia L.* are known for its inherent antimicrobial properties [9] and incorporation of this oil into the resin system will provide excellent resistance against microbial activity [9, 10]. The hydroxyl functionality of these oils make them natural polyol, providing a longer shelf life than other oils by preventing peroxide formation, and serve as a reaction site for various chemical reactions [1, 11]. Furthermore, these properties make alkyd resin to be useful in coatings, lubricants, paints, and in industrial and domestic applications [12]. This shift towards sustainable materials aligns with global efforts to reduce environmental impact while maintaining the high-performance characteristics required in antimicrobial coatings [12, 13].

The composition and the process variation during the synthesis of alkyd resins may have significant effect on the application properties of alkyd resins. Therefore, it is important to understand the physical characteristic changes of the reaction during the synthesis of alkyd resins via kinetics [14].

Whereas several studies [10-14] have explored alkyd resins derived from vegetable oils and their nanoparticle modifications, few have incorporated both garlic extract and multiple metal oxide nanoparticles synergistically. This research seeks to synthesize alkyd resins from non-edible castor and walnut seed oils, to formulate eco-friendly low-solvent coatings; and enhance their anti-bacterial protective properties through nanoparticle-plant extract incorporation.

This study uniquely demonstrates the combined antibacterial effects of CuO/ZnO/TiO_2 nanoparticles and garlic extract in castor and walnut oil-derived alkyd resins, addressing gaps in prolonged antimicrobial efficacy and bio-based formulations.

MATERIALS AND METHODS

All reagents and solvents used were of analytical grade and were used as received without further purification

Seeds Collection and Preparation

Non-edible castor and walnut seeds, with the view of obtaining crude castor oil (CCO) and crude walnut oil (CWO), were sourced from farms and markets in Ilorin, Kwara State, Nigeria,

Oil Extraction and Refining

The oils were extracted using n-hexane following the method of Olaoluwa *et al*. [15], with slight modifications. Approximately 40 g of the dried sample was soaked in 200 mL of n-hexane, stirred at 300 rpm for 20 hours at 45°C. The mouth of the beaker was tightly closed with aluminum foil to prevent the evaporation of solvent. After extraction, the hexane solution was filtered using 125 mm grade filter paper (ChM lab group, Barcelona, Spain) and then evaporated in a rotary vacuum evaporator at 40°C. The extracted oil was refrigerated until analysis. Oil extracted by n-hexane was considered as total oil and the refinement processes prior to trans-esterification. involved degumming, alkaline and bleaching treatment which were conducted by following the method of Olaoluwa *et al* [15], The % yield of the obtained oils was calculated using Equation 1:

$$Oil\ yield\ (\%) = \frac{mass\ of\ oil\ produced}{mass\ of\ seed\ used} \times 100 \qquad (1)$$

Physicochemical Characterization of the Seed Oil

Physicochemical properties of the seed oils extract such as colour odour, density, viscosity, specific gravity and refractive index, acid value (mg KOH/g), iodine value (mg I_2/100g), peroxide value (meq O_2/kg), saponification value (mg KOH/g), and free fatty acids were determined by following AOAC methods [16-18].

Synthesis of Alkyd Resin

The procedure for alkyd resin synthesis involved two stages: alcoholysis and esterification.

Alcoholysis stage

The apparatus were sets, 120 g of each oil of samples were charged into the reactor with 0.2 g of CaO as catalyst. The mixture was heated up to about 120°C, after which 31.881 g of castor or walnut and 31.85 g of glycerol was added and heated up to 230-250°C. This is maintained for 1h 30 minutes with vigorous agitation, resulting in the trans esterification of triglyceride into a mixture of mono- and diglyceride oils. Alcoholysis process was completed when the sample of the mixtures formed became soluble in 1 to 3 mL of anhydrous methanol to give a clear solution. After this, the reaction temperature was cooled to 120°C.

Esterification stage

For esterification process, the temperature was set to 120°C after 30 minutes, 48.119 g of castor or walnut with 48.155 g of phthalic anhydride were added to the reaction mixture, followed by addition of xylene (10% of total weight charged) into the reaction mixture. The water from esterification formed an azeotrope with xylene, which was removed at intervals. Then, the temperature was increased to 245°C, with continuous stirring to build the molecular weight of the resin [19, 32]. Xylene was added to remove the water produced as by-product raising the temperature to above 250°C with continuous stirring. The reaction was monitored by periodically checking the acid number and viscosity after 180 min. The process was stopped when the solution was fairly viscous and acid value found below 10. Then, it was partially cooled and poured into storage container, for further study. The acid value of in-process samples taken at intervals were determined by titrating with a 0.1M KOH solution to the phenolphthalein end point after dissolution in a mixture of toluene and ethanol (1:1) [19]. The acid value is related to the extent of the reaction as calculated from Equations 2 and 3:

$$T = P_{AV} = \frac{AV_o - AV_t}{AV_O} \quad (2)$$

Or

$$\frac{AV_t}{AV_o} = 1 - P \quad (3)$$

The average degree of polymerization Dp as given in Equation 4:

$$D_p = \frac{\text{no of molecules originally present}}{\text{no of molecules finally present}} \quad (4)$$

$$\frac{AV_o}{AV_t} = (1 - P)^{-1} \quad (5)$$

$$\frac{AV_o}{AV_t} = AV_o kt + constant \quad (6)$$

Where AV_o is the initial acid value, AV_t is the acid value after time, t, of the reaction, k is the rate constant and t is the time of reaction [20]. A plot of AV_o/AV_t gives a straight line AVo.k from which k can be obtained.

Physicochemical Characterization of the Synthesized Alkyd Resin

Structural Analysis by FTIR

FTIR spectra were obtained using a Shimadzu spectrophotometer with samples prepared as KBr pellets. Spectral scans were conducted in the range of 4000–400 cm^{-1} to identify functional groups in the seed oils.

Fatty Acid Profiles using GCMS

GC-MS analysis was conducted using Varian 3800 gas –chromatography coupled with Agilent MS capillary column (30 m x 0.25 mm). The equipment was connected to Varian 4000 Mass Spectrophotometer (EI mode 70eV: m/2/ -1000) source temperature 230^{O}C and quadruple temperature of 150^{O}C. The column temperature was initially at 200^{O}C (2 min) and raised to 300^{O}C (4 min). Nitrogen (carrier) gas flow rate was set 1.0 mL/min. The oil sample (1 mL) was mixed with chloroform and injected using 50:1 split ratio. The mass spectrophotometer was set to scan the sample in the range of m/z 1 - 1000 with electron impact ionization mode. Both samples, refined castor oil (RCO) and refined walnut oil (RWO), were analyzed as one batch. Prepared pooled sample which was injected at regular intervals was used as control to provide set of data for repeatability assessment. GC-MS was conducted according to AOAC Official Method [18] with interpretation guided by the NIST v2.1 MS data library.

Green Modification Alkyd Resin using Metal Oxide Nanoparticles

Nanoparticle-Modified Alkyd Resin

The nanoparticles (CuO, ZnO and TiO_2) were dispersed into the alkyd resin matrix at 2% g/g and 4% g/g using a high-shear mechanical stirrer at 1200 rpm for 30 minutes at room temperature. Uniform dispersion was ensured through sonication for 10 minutes as NP-alkyd.

Garlic-Enhanced Nanoparticle-Modified Alkyd Resin

Garlic extract was introduced during the nanoparticle synthesis phase as a bio-reducing and capping agent. The resulting garlic-functionalized nanoparticles were incorporated into the alkyd resin matrix using the same dispersion method as described above. This formulation is referred to as G-NP-alkyd. The synthesis protocol for garlic-modified nanoparticles was adapted from Shankar and Rhim [65], ensuring a green synthesis approach and uniform nanoparticle distribution within the resin.

Antibacterial testing

The coating formulations' antimicrobial efficacy was tested per the JIS Z 2801 assay. The Alkyd resin was tested against both bacterial strains used were *Escherichia coli* (Gram-negative), *Staphylococcus aureus* (Gram-positive), and *Pseudomonas aeruginosa*. These were obtained from a microbiology culture collection and maintained on nutrient agar slants. Overnight cultures were prepared in nutrient broth and adjusted to a 0.5 McFarland standard (1.5×10^8 CFU/mL). After incubation, the diameter of the inhibition zones (including the disc diameter) was measured in millimeters using a digital veneer caliper. Each test was performed in triplicate, and the mean ± standard deviation was recorded [21].

RESULTS AND DISCUSSION

Physicochemical Properties of the Seed Oils

Vegetable oils are crucial in various industrial, pharmaceutical, and food applications due to their diverse physicochemical properties. These properties, including viscosity, density, refractive index, acid value, iodine value, and saponification value, are largely dictated by the fatty acid composition, degree of unsaturation, and refining processes [22, 23]. Table 1 shows the physicochemical characteristics of castor and walnut seed oils in both crude and refined states, emphasizing industrial relevance and optimization through refining processes.

Table 1. The physiochemical properties of the seed oils

Physicochemical properties	Refined Vegetable Oils	
	Castor oil (RCO)	Walnut oil (RWO)
Yield (%)	32.0	56.3
Refractive Index	1.49	1.51
Density at 40°C (g/cm^3)	0.94	0.91
Viscosity (cp, 30 °C)	202	54
Acid value, (mg/KOH)	1.2	0.8
Saponification,(mg/KOH)	196.39	185.47
Iodine Value, (I_2/100g)	79	128
%FFA	0.6	0.4

The densities of the oils were 0.94 and 0.91 for castor and walnut seed oils respectively, which were less dense than water.

The iodine values were 79 and 128 g I_2/100 g for RCO and RWO respectively, which implies that both oils are semi-drying oil. The iodine value is an important parameter employed in ascertaining the suitability of oil for alkyd synthesis. It implies that the walnut is a semi-drying oil whose iodine value is in the range of 120 - 150 g I_2/100 g [24]. Semi-drying oils have various applications such as in the synthesis of alkyd resins for the paint industry and in the manufacturing of soap, whereas the non-drying oils are majorly employed as plasticizers [25].

The saponification value obtained for the oils were 196.39 and 185.47 mg KOH/g for castor and walnut seed oil respectively, and this suggests that both oils may be suitable for the manufacture of soap due to its high molecular weight fatty acid [26].

The acid value of the both oils were found to be 1.2 and 0.8 mg KOH/g for castor and walnut seed oil respectively, which suggests low activity of enzymes in the seed which also denotes its stability [27] and therefore enhances its industrial application in the manufacture of paints and varnishes at low acid values [28]. Acid value is used to measure the level of deterioration of oil. It has been reported that high acid value of oil could be due to hydrolytic reaction during processing of the oil or as a result of enzymatic action in the oil-bearing seed [29]. The refractive indexes for the oils were 1.49 and 1.51 for castor and walnut seed oil respectively, and are within the range of 1.460 - 1.540, which suggests that it can serve as a test for purity and a means for identification [30].

The physicochemical properties of vegetable oils vary significantly depending on their composition and refining status. Refining improves stability by reducing FFAs, lowering iodine values, and modifying viscosity, enhancing usability. Among the studied oils, castor oil exhibits the highest viscosity, making it ideal for lubricants and industrial fluids. Walnut oil has the highest iodine value, reflecting its high PUFA content, which is nutritionally beneficial but predisposes it to oxidation.

The findings underscore the critical role of refining in optimizing vegetable oils for specific applications, ensuring their stability and efficiency across industries [23, 31,32].

Fatty Acid profiles

Table 2 shows the fatty acid composition. It influences their industrial alkyd applications. The oils, as feedstock, are known for its high hydroxylases fatty acid content. It plays a crucial role in alkyd resin synthesis, where its fatty acid composition affects film formation, drying properties, and polymer reactivity [33]. The % oils saturation and unsaturation were 13.11 and 85.60% respectively for castor oil, and 12.02 and 86.97% respectively for walnut oil. It modifies the fatty acid profile, impacting viscosity, oxidative stability, and saponification value, which are essential in alkyd resin production [33].

Table 2. Fatty acids profile of crude and refined castor oils

Refined Castor Oil (RCO)			Refined Walnut Oil (RWO)		
Fatty acid	Saturation	Composition (%)	Fatty acid	Saturation	Composition (%)
Palmitic Acid	$C_{16}H_{32}O_2$	7.10	Palmitic acid	$C_{16}H_{32}O_2$	7.10
Stearic Acid	$C_{18}H_{36}O_2$	4.16	Palmiloleic	$C_{16}H_{30}O_2$	0.09
Oleic Acid	$C_{18}H_{34}O_2$	35.03	acid	$C_{18}H_{36}O_2$	4.16
Linoleic Acid	$C_{18}H_{32}O_2$	50.57	Stearic acid	$C_{18}H_{34}O_2$	35.03
Arachidic Acid	$C_{20}H_{40}O_2$	0.33	Oleic acid	$C_{18}H_{32}O_2$	50.57
Cis-11-			Linoleic acid	$C_{20}H_{40}O_2$	0.33
Eicosenoic Acid	$C_{20}H_{38}O_2$	0.61	Arachidic acid	$C_{18}H_{30}O_2$	0.67
Heneicosanoic			Linolenic acid	$C_{20}H_{38}O_2$	0.61
Acid	$C_{21}H_{42}O_2$	0.34	cis-11-	$C_{24}H_{48}O_2$	0.43
Behenic Acid	$C_{22}H_{44}O_2$	0.75	Eicosenoic		
Lignoceric Acid	$C_{24}H_{48}O_2$	0.43	acid		
% Saturation		13.11	Lignoceric		
% Unsaturation		85.60	acid		
			% Saturation		12.02
			%		
			Unsaturation		86.97

Palmitic and stearic acid accounts for 7.10 and 4.16% respectively for castor oil, and 7.10 and 4.16% respectively, for walnut oil. This influences the resin's flexibility, as SFAs enhance intermolecular interactions, increasing hardness and brittleness in coatings [34]. Consequently, a controlled SFA content is necessary to balance durability and flexibility in alkyd resin formulations.

The oleic acid content was 35.03%, for both oil. This enhancement improves oxidative stability and reduces lipid peroxidation, which is crucial for maintaining resin longevity [35],

Linoleic acid (C18:2), a major polyunsaturated fatty acid (PUFA), content was 50.57% in the both oil. Higher PUFA content is advantageous in alkyd resin synthesis as it enhances drying properties and cross-linking potential upon polymerization [35]. However, excessive PUFA levels can increase susceptibility to oxidation. Other fatty acid present in the oils including linolenic, arachidic, eicsinoic acid among others are presented in Table 2.

FT-IR analysis of the Seed Oils

Fourier transforms infra-red spectra of the extracted seed oils (castor and walnut) were obtained using FT-IR (ATR- FTIR) in order to identify the functional groups present as presented in Figures 1 and 2.

Castor oil – FTIR (KBr, cm^{-1}): -O-H (3441 cm^{-1}), C-H (2922 cm^{-1}), -COO- (2348 cm^{-1}), C=O (1664 cm^{-1}), C=C (1640 cm^{-1}), C-H bending (877 cm^{-1}) and –C-O (1057 cm^{-1}).

Walnut oil – FTIR (KBr, cm^{-1}): -O-H (3764 & 3425 cm^{-1}), C-H (2923 & 2857 cm^{-1}), -COO- (2357 cm^{-1}), C=O (1627 cm^{-1}), C=C (1627 cm^{-1}), CH_2 & CH_3 bending (1382 and 1316 cm^{-1}), –C-O (1022 and 1087 cm^{-1}) and C-H bending (608 cm^{-1})

The FTIR spectra confirms the presence of triglycerides and fatty acids. The main functional groups identified correspond to saturated and unsaturated fatty acids found in castor and walnut oil's composition. Since the extracted castor and walnut oil were composed of essential fatty acid and esters; it was observed that their FT-IR spectra were found at similar frequencies of absorption; the O-H carboxylic acid stretch which was observed at the 3441 cm^{-1} in the castor oil was seen at the 3425 cm^{-1} in the walnut oil spectrum. The appearance of broad absorption peaks in this region suggests the presence of hydroxyl compounds or residual water in the oil [36]. The -CH_2 alkane stretching which was observed at the 2922 cm^{-1} in the castor oil, was observed at the 2923 cm^{-1} in the walnut oil spectrum. These peaks

confirm the presence of saturated and unsaturated fatty acid. The stretching vibrations arise from the C-H bonds in the aliphatic chains of the fatty acids [37].

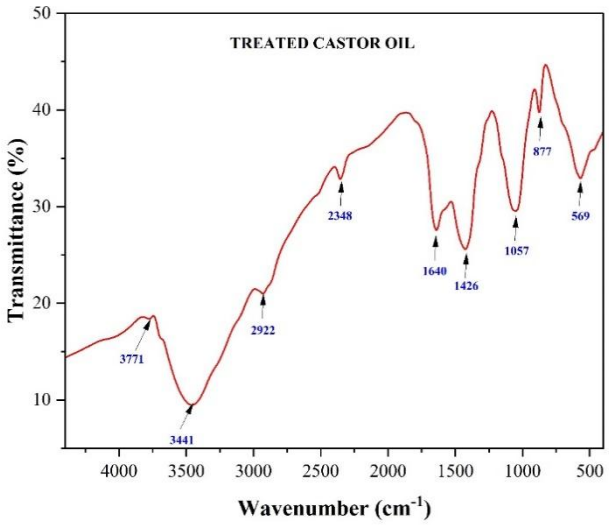


Figure 1: FTIR result of refined castor oil

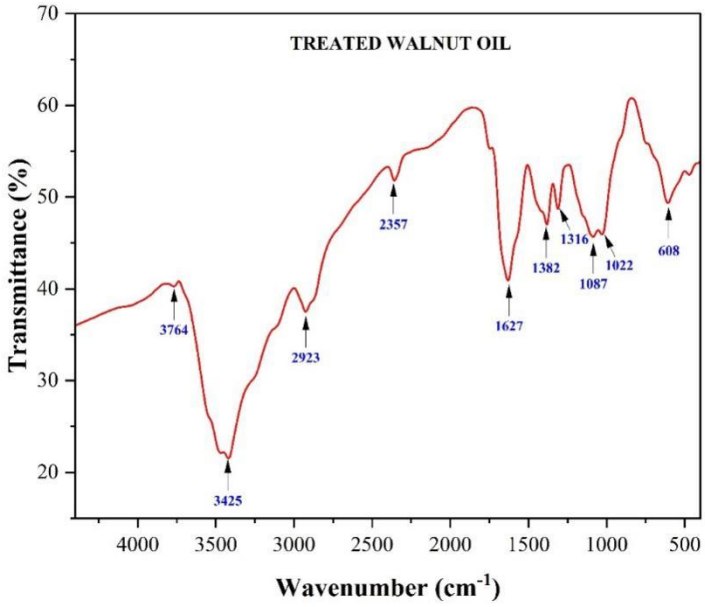


Figure 2. FTIR result of refined walnut oil

The C=O stretching of a carboxylic and triglycerides (ester) which was observed at the 1664 cm^{-1} in the castor oil was observed at the 1627 cm^{-1} in the walnut oil spectrum.

Another similarity was noted in their C=C unsaturated stretch which was found at 1640 cm^{-1} in the castor oil and occurring at 1627 cm^{-1} in walnut oil spectrum, which is characteristic of unsaturated fatty acids [38].

However, both oils possessing a strong, broad O-H stretch of alcohol at the 3441 cm^{-1} and 3425 cm^{-1} which could be attributed to the presence of an intermolecular bond between the fatty acids.

It was also observed that there was a strong, broad CO- stretching of a tertiary alcohol which was around 1022 and 1057 cm^{-1} for castor and walnut oil spectra respectively. These peaks confirm the presence of ester (-C-O) groups, which are characteristic of triglycerides in both oils [39]. This confirms that both oils are rich in triglycerides rather than free fatty acids.

Alkyd Resins

Table 3 and Figure 3a-d, show the synthesis of alkyd resins from oils. It involves several key parameters used to assess the completion of the reaction and the properties of the resulting resin. The key parameters that were analyzed include the acid value, degree of polymerization (Dp), conversion of functional groups (P), and the percentage conversion of functional groups (%P). These parameters are crucial for understanding the chemical transformations during alkyd resin synthesis.

Acid Value analysis

At $T_{0.0}$ (the initial stage of the reaction), the acid value of castor and walnut oils were found to be 37.59 ± 0.40 and 34.21±6.07 mg KOH/g, respectively, which are typical for oils before undergoing esterification or polymerization [40]. As the reaction progressed, the acid value decreased, signifying the conversion of free fatty acids into esterified compounds. At T_{30}, the acid value decreased to 18.86 ± 0.22 and 22.81±0.59 mg KOH/g, for castor and walnut oil respectively. This trend continued throughout the reaction. At the final stage of reaction, T_{120}, the acid value reached 8.84 ± 0.01 and 8.18±0.08 mg KOH /g, for castor and walnut oil respectively. The final acid value of around 8.84 mg KOH/g is consistent with values reported for other alkyd resins synthesized from similar oils [41], suggesting that the reaction conditions used in this synthesis were effective in producing a stable resin with low reactivity and good film-forming properties. This decrease in acid value of the seed oils improves the resin's stability and reduces reactivity, which is crucial for applications such as coatings [42], as presented in Figure 3 and Table 3.

Table 3. Acid value of long castor and walnut alkyd resin

S/N	Reaction time	Castor alkyd resin				Walnut alkyd resin			
		Acid value	Dp	P	%P	Acid value	Dp	P	%P
1	$T_{0.0}$	37.59±0.40	-	-	-	34.21±6.07	-	-	-
2	T_{30}	18.86±0.22	1.99	0.50	50	22.81±0.59	1.50	0.33	33
3	T_{60}	16.98±3.05	2.21	0.55	55	14.64±1.12	2.34	0.57	57
4	T_{90}	11.78±0.42	3.19	0.69	69	12.94±1.10	2.64	0.62	62
5	T_{120}	8.84±0.01	4.25	0.77	77	8.18±0.08	4.18	0.76	76

(Dp) degree of polymerization, (P) conversion of functional groups, (%P) percentage conversion of functional groups.

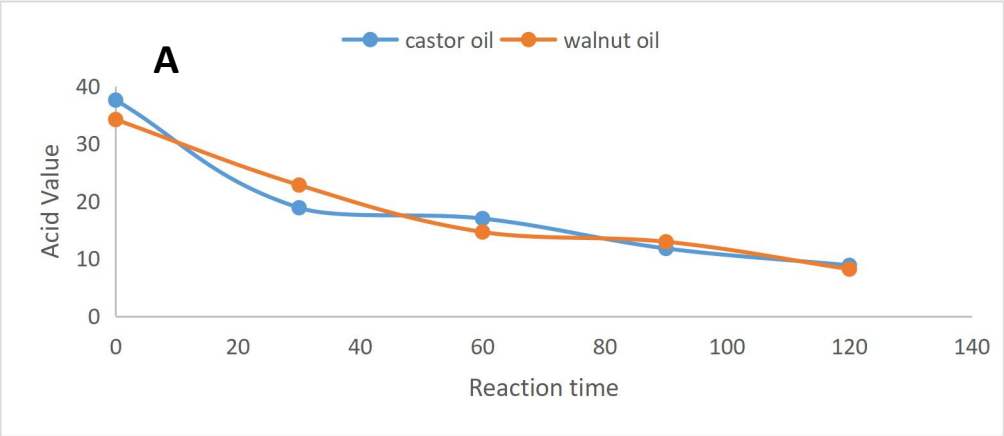
Degree of polymerization (Dp)

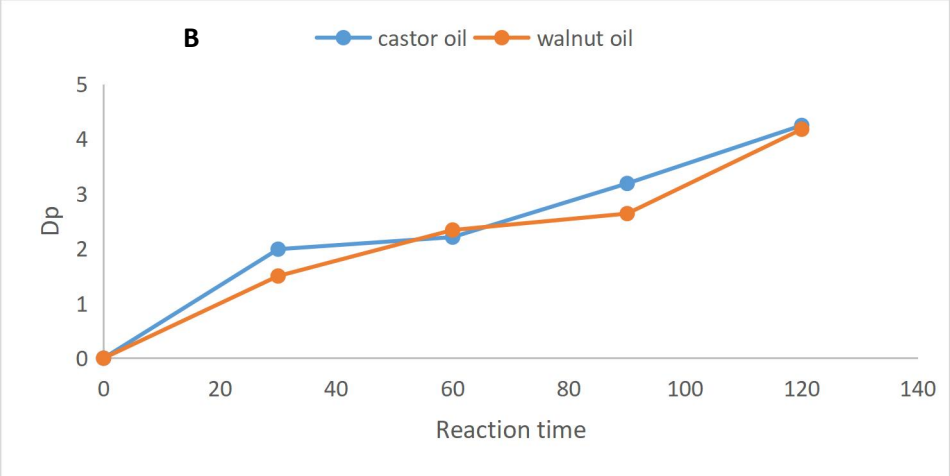
At initial $T_{0.0}$, no polymerization occurred, the Dp is not measurable. However, as the reaction progressed, the Dp increased, reflecting the development of polymer chains. At T_{30}, the Dp is 1.99 and 1.50 for castor and walnut oil respectively, and it increased gradually to 4.25 and 4.18 for castor and walnut oil respectively, at T_{120}. This increase in Dp indicates that polymerization is occurring as the resin forms, and the longer reaction times lead to the creation of larger polymer chains and a more cross-linked structure. The trend in Dp over time is consistent with the general understanding of alkyd resin synthesis, where esterification and polymerization are integral to the formation of the resin [43], as presented in Figure 3b and Table 3.

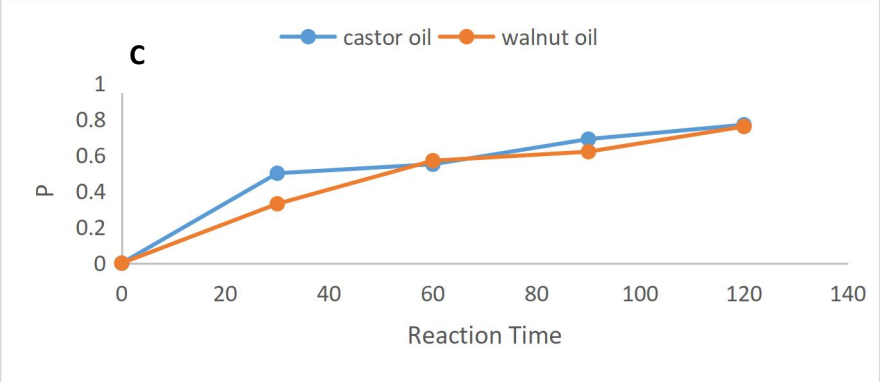
Conversion of Functional Groups (P)

At initial stage $T_{0.0}$, there's no functional group conversion. As the reaction proceeded to T_{30}, the conversion of functional groups (P) is 0.50 and 0.33 for castor and walnut oil respectively, indicating that a small fraction of the functional groups has been converted into ester bonds. As the reaction continued, the conversion of functional groups increased, reaching 0.77 and 0.76 for castor and walnut oil respectively, at T_{120}. The steady increase in the conversion of functional groups over time supports the notion that esterification and polymerization are progressing as expected, with functional groups progressively being transformed into ester

bonds [42]. This gradual increase in P suggests that the synthesis is moving toward the completion of a fully functional resin structure, as presented in Figure 3c and Table 3.







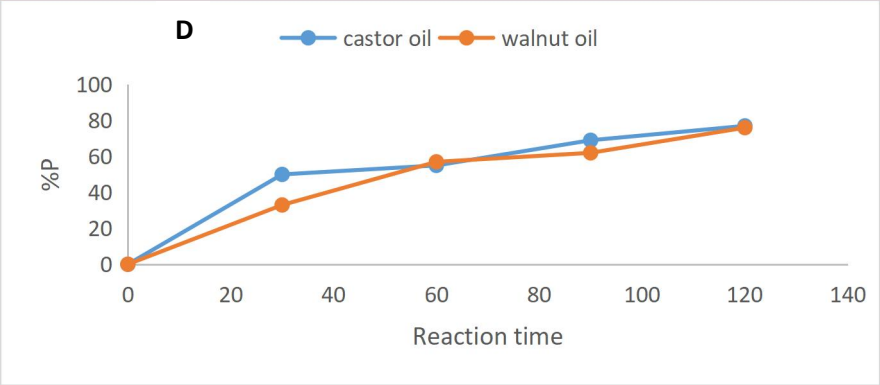


Figure 3. In-situ characterization of alkyd resin synthesis: A). Acid value, B). Degree of polymerization (Dp), C). Conversion of functional groups (P) and D). Percentage conversion of functional groups (% P).

Percentage Conversion of Functional Groups (% P)

At initial stage $T_{0.0}$, there was no functional group conversion. As the reaction proceeded to T_{30}, the percentage conversion of functional groups is 50 and 33 % for castor and walnut oil respectively, and it steadily increased, reaching 77 and 76 % at T_{120}, for castor and walnut oil respectively. This gradual increase indicates that a significant portion of the functional groups in seed oils has been successfully converted into ester bonds, thereby forming the alkyd resin structure [44]. The trends observed in this data align well with findings from the literature report on alkyd resins synthesized from vegetable oils [45, 46]. For instance, the reduction in acid value over time, as well as the increase in Dp and %P, are typical indicators of successful esterification and polymerization, which are essential steps in alkyd resin synthesis. Furthermore, the final acid value of 8.84 mg KOH/g is consistent with values reported for other alkyd resins synthesized from similar oils [41], suggesting that the reaction conditions used in this synthesis were effective in producing a stable resin with low reactivity and good film-forming properties, as presented in Figure 3d and Table 3

GC-MS of Alkyd Resin Synthesis from the Seed Oils

The GC-MS analysis of the alkyd resin synthesized from refined castor and walnut oil reveals a complex profile of organic compounds, some of which are direct derivatives of the fatty acids present in the oils, as presented in Figures 3 and 4.

Phthalic anhydride is a major component in the castor oil with peak area of 17.01% and 0.83%. This compound is a key reactant in alkyd resin synthesis, provides aromatic rings that contribute to hardness, chemical resistance, and rigidity which are essential properties in

coatings. The significant presence in the GC-MS spectrum reflects its incorporation into the alkyd backbone through esterification with fatty acids like oleic and linoleic acid [47].

9,12-octadecadienoic Acid (Z,Z) has a peak area of 7.55%, 3.35%, 0.92%, and 0.94% in castor oil and peak area of 16.25 %, in walnut oil. This peak corresponds to linoleic acid, in facilitating rapid curing [48].

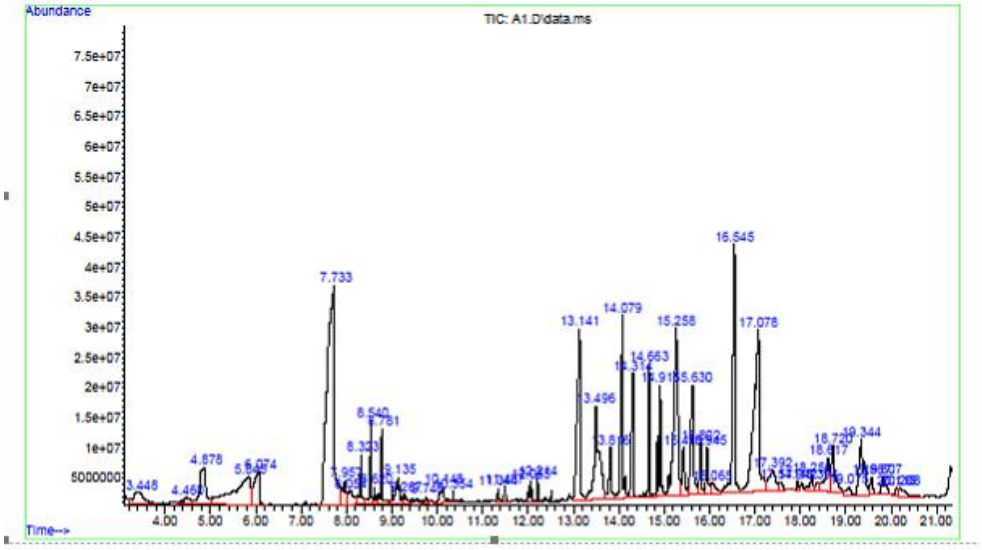


Figure 4. GC-MS chromatogram of Castor Alkyd Resin

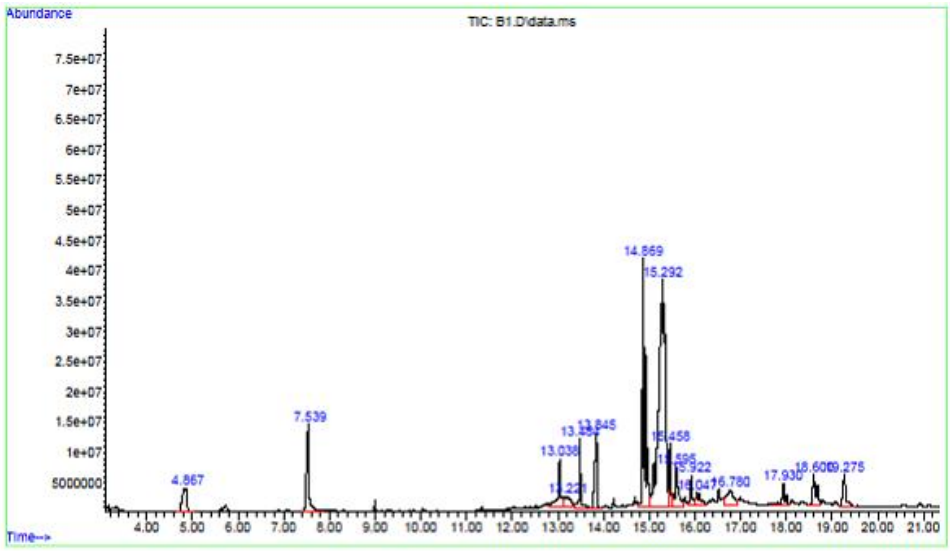


Figure 5. GC-MS chromatogram of Walnut Alkyd Resin

Ricinoleic acid has a peak area of 9.40% in castor oil. The significant presence in the alkyd resin highlights its contribution to the resin's adhesion to substrates, which is crucial for adhesive applications [49].

Hexadecanoic acid has a peak area of 4.81% in castor oil and peak area of 4.32% in walnut oil. Hexadecanoic acid (palmitic acid), is a saturated fatty acid found in oils. Its presence also helps control the viscosity and film-forming properties, making the resin more suitable for high-performance coatings [41].

Other minor components includes: 10-Undecenoic Acid, Methyl Ester: (0.25 %), 9,12-Octadecadienoic Acid (Z,Z)- (E,E)-Methyl Ester (2.22 %), Hexadecanoic Acid, 2-Hydroxy-1-(Hydroxymethyl)Ethyl Ester (2.87 %). This ester of hexadecanoic acid with a hydroxy group enhances the polarity of the resin, improving its adhesion properties. This is particularly valuable for coatings that require strong bonding to substrates [50]

FTIR Analysis of Alkyd Resin

The FTIR spectra confirms the successful synthesis of alkyd resin from castor and walnut oil.

Castor-Alkyd resin – FTIR (KBr, cm^{-1}): -O-H (3432, 3538, 3786 cm^{-1}), C-H (2925 cm^{-1}), C=O (1740–1730 cm^{-1}), C=C (1599 cm^{-1}), C-H bending (1424 & 1331 cm^{-1}) and –C-O-C (1043 cm^{-1}).

Walnut-Alkyd resin – FTIR (KBr, cm^{-1}): -O-H (3439 cm^{-1}), C-H (2921 cm^{-1}), C=O (1640 cm^{-1}), C=C (1640 cm^{-1}), –C-O (1035 cm^{-1}) and C-H bending (589 cm^{-1})

The O–H stretch suggests incomplete esterification or the retention of hydroxyl groups from castor and walnut oil's unique ricinoleic acid composition, aligning with a previous study [51]. The presence of ester (C=O, C–O–C) peaks confirms the esterification reaction between fatty acids and polyols, supporting previous findings [52, 53]. The C=C peak (1599 cm^{-1}) suggests that unsaturated fatty acids from RCO (especially linoleic and oleic acids) are still present, influencing drying properties, consistent with previous studies [54]. C=C stretching at 1640 cm^{-1} suggests retained unsaturation, consistent with walnut oil-based alkyds studies [54, 55].

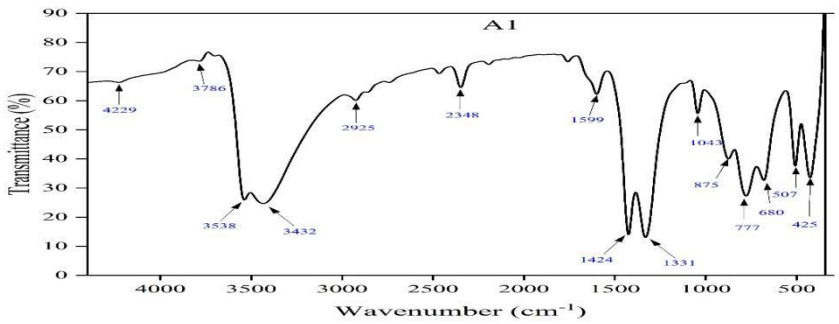


Figure 6. FTIR spectrum of castor Alkyd resin

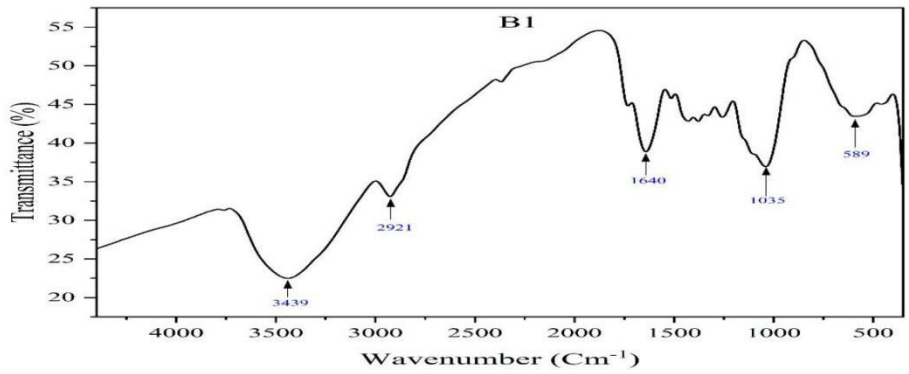


Figure 7. FTIR spectrum of walnut Alkyd resin

The presence of skeletal vibrations (589 cm^{-1}) is in agreement with researchers [56], who studied green alkyd coatings with antimicrobial properties. The FTIR spectrum of alkyd resin confirms the successful synthesis of alkyd resin from castor and walnut oil, with clear evidence of esterification (1035 and 1043 cm^{-1}) and retained unsaturation (1640, 1740–1730 cm^{-1}). These findings align with studies on walnut oil-based alkyds for coatings, corrosion resistance, and enhanced mechanical properties [51, 57, 58]

Antibacterial analysis

Antibacterial Activities of Alkyd Resins

Table 4 presents the antibacterial activities of alkyd resins against test Isolates alkyd resins. The study evaluates the antibacterial activities of different alkyd resins against *Staphylococcus aureus* (Gram-positive), *Escherichia coli*, and *Pseudomonas aeruginosa* (both Gram-negative), using the zone of inhibition (ZOI) method. The seed oils exhibited susceptibility against *S. aureus* at 22.5 and 25 mm respectively, intermediate inhibition for *E. coli* at 10 and 12.5 mm, for castor and walnut oil respectively and resistance against *P. aeruginosa* for castor oil, and against at 11 mm for walnut oil. Castor oil contains ricinoleic acid, which has well-documented antibacterial activity [59].

Table 4. Antibacterial activities of Alkyd resins against test isolate.

Alkyd resin	(Zone diameter of inhibition in mm)		
	S. aureus	*E. coli*	*P. aeruginosa*
Castor	22.5	10	R
Walnut	25	12.5	11
Susceptible (≥ 20 mm zone of inhibition), Intermediate (≥10-20 mm zone of inhibition),Resistant (R): (≤ 10 mm zone of inhibition).			

The reduced activity against *P. aeruginosa* could be due to its intrinsic resistance mechanisms, such as efflux pumps and low membrane permeability [60]. However, its susceptibility to *S. aureus* confirms earlier studies on the effectiveness of castor oil-based polymers against Gram-positive bacteria [61]. The strong inhibition of *S. aureus* aligns with previous findings that walnut oil extracts are effective against Gram-positive bacteria [62]. However, *P. aeruginosa* remained resistant, likely due to its advanced resistance mechanisms, including biofilm formation and efflux systems [60].

Control Study

The study highlights the antibacterial potential of garlic extract against three significant pathogens: *Pseudomonas aeruginosa*, *Escherichia coli*, and *Staphylococcus aureus*, using Ciprofloxacin (50 mg/ml) as control.

Table 5. Antibacterial activities of garlic extract

Isolates	Garlic Extracts 100 mg/ml	Ciprofloxacin (50 mg/ml)
Pseudomonas aeruginosa	28.33±5.7	32.2±1.14
Escherichia coli	35.0±5.00	37.5±3.54
Staphylococcus aureus	32.50±3.54	35±1.02

Data is expressed as mean ± Standard Error with a significant difference of ($P < 0.05$). control: Ciprofloxacin (50 mg/ml).

Pseudomonas aeruginosa*:* The garlic extract showed a mean zone of inhibition of 28.33±5.7 mm, slightly less than the control, ciprofloxacin at 32.2±1.14 mm. This suggests a notable antibacterial activity but indicates that ciprofloxacin remains more effective under the study's conditions.

Escherichia coli*:* Garlic extract exhibited a strong inhibition zone of 35.0±5.00 mm, approaching ciprofloxacin's 37.5±3.54 mm. This positions garlic as a robust alternative or complement in managing infections caused by *E. coli*.

Staphylococcus aureus*:* The inhibition zone of garlic extract was 32.50±3.54 mm compared to ciprofloxacin's 35±1.02 mm, showing its significant effectiveness against Gram-positive bacteria.

Antibacterial Analysis of Garlic-Nanomaterial Modified Oil-Alkyd Resin

Table 6 reveals the potential of garlic-nanomaterial modified oil-alkyd resin formulations as effective antibacterial agents against *Staphylococcus aureus, Escherichia coli* and *Pseudomonas aeruginosa*

Staphylococcus aureus*:* The Metal Oxide-NPs incorporated into castor oil-alkyd resins displayed the highest antibacterial activity of CuO/TiO_2 (48.5 ± 3.54 mm) and $CuO/ZnO/TiO_2$ (47 ± 9.90 mm) against ***S. aureus*****,** indicating that the oil formulations are ideal for prolonged action. While for walnut oil-alkyd resins, ZnO/TiO_2 exhibited the highest antibacterial activity, with a ZOI of 45 ± 14.14 mm. CuO/ZnO and $CuO/ZnO/TiO_2$ also showed susceptibility with ZOIs of 25 ± 8.49 mm and 29.5 ± 7.78 mm, respectively [63]. The hydrophobic nature of oils may facilitate sustained release of garlic-modified nanoparticles, enhancing efficacy [64].

Escherichia coli**:** The NP-garlic extract incorporated into castor and walnut oil-alkyd resins against E. coil exhibited a moderately high inhibition zone of CuO/ZnO (34.5±21.92 & 22.5±12.02 mm) respectively, indicating that garlic extract can still improve antibacterial activity even in more complex formulations.

P. aeruginosa: For *P. aeruginosa*, CuO/ZnO/TiO2 nano-composite produced a significant inhibition zone (41.5 mm), highlighting the synergistic effects of garlic extract and metal oxide nanoparticles in castor-based resins. The CuO/ZnO nano-composite in walnut-based resin displayed a substantial inhibition zone of (31 mm), this aligns with studies suggesting that combined nanoparticle formulations are more effective against bacteria [63]. The global rise of antibiotic-resistant bacterial has created a demand for innovative antimicrobial strategies. Metal oxide nanoparticles (MO-NPs) are a promising alternative due to their high surface area, bioactivity, and ability to generate reactive oxygen species (ROS) [65]. Garlic (*Allium sativum*) extract, known for its antimicrobial compounds such as allicin, can further enhance the efficacy of MO-NPs when incorporated into oil-alkyd resins [65].

Table 6. Antibacterial activities of oil-alkyd resin with metal oxide nanoparticles modified with garlic extract against *E. coli, P. aeruginosa and Staphylococcus aureus*

Alkyd resin	Extracts at 100mg/ml (zone diameter of inhibition in mm)							
		CuO	ZnO	TiO_2	CuO/TiO_2	ZnO/TiO_2	CuO/ZnO	$CuO/ZnO/TiO_2$
Castor	*E. coli*	16±12.73	10±1.21	11±12.73	13.5±3.44	34.5±21.92	17.5±4.75	11±12.73
	P. aeruginosa	35±0.71	32.5±3.54	20±7.07	41.5±3.54	40±28.28	30.5±0.71	27±9.90
	S. aureus	3.5±0.71	42.5±3.54	40±7.07	48.5±3.54	40±28.28	40.5±0.71	47±9.90
Walnut	*E. coli*	22.5±3.54	28±16.97	16.5±6.36	22±11.31	22.5±12.02	24.5±2.12	18.5±7.78
	P. aeruginosa	25±7.07	29.5±3.54	22.5±10.61	31±1.41	35±14.14	31±8.49	27±7.78
	S. aureus	25±7.07	29.5±3.54	22.5±10.61	31±1.41	45±14.14	25±8.49	29.5±7.78

Interpretation: Susceptible (≥ 20 mm zone of inhibition), Intermediate (≥10-20 mm zone of inhibition), Resistant: (≤ 10 mm zone of inhibition).

CONCLUSION

This study involved the extraction of oils from castor and walnut seeds, the synthesis and physicochemical characterization of the resulting alkyd resins, and the evaluation of their antibacterial performance when modified with garlic extract-functionalized metal oxide nanoparticles. Walnut oil displayed superior raw properties namely low acid value and high iodine value indicating excellent chemical stability and drying potential for high-performance alkyd resins. Although castor oil exhibited less favorable initial characteristics, its conversion into a long oil alkyd resin produced a polymer with strong physicochemical attributes, including a final acid value of 8.84 mg KOH/g and a functional group conversion rate of 77%. The walnut alkyd resin achieved a lower acid value (8.18 mg KOH/g) and a comparable conversion rate (76%). Incorporating garlic-modified nanoparticles (CuO, ZnO, TiO_2) significantly improved the antimicrobial performance of both resins, with the castor-based resin showing the highest inhibition zone (49.5 mm). The walnut oil-based medium alkyd resin also demonstrated strong antibacterial activity, especially when modified with ZnO/TiO_2 and CuO/ZnO combinations. It also provides other superior antimicrobial properties than the castor oil-derived alkyd resin, when properly nano-composite-modified. These findings underscore the potential of bio-based alkyd resins, particularly those enhanced with garlic-modified nanomaterials, as promising candidates for antimicrobial coatings in sustainable smart-city and healthcare applications.

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