SYNTHESIS AND CHARACTERIZATION OF ALKYD RESIN IMMERSED IN SILVER-NANOPARTICLE DERIVED FROM COTTONSEED OIL

Abstract

Alkyd resin prepared from *Gossypium hirsutum*(cotton seed) seed oil and immersed in silver nanoparticles has been characterized and evaluated using standard analytical methods. Gas Chromatography-Mass Spectrometry(GCMS) was used to determine the fatty acid profile of the seed oil extractwhich was physicochemically characterized using AOAC standard method.The resultsobtained are as follows: Iodine value;102.36gI_2/100g, Acid value; 3.19mgKOH/g,Saponification value; 202.32 mg/KOH/g, Refractive index;1.46, Specific gravity; 0.91and Moisture content; 1.20. The alkyd resin was prepared using alcoholysis-polyesterificationreaction. The prepared resin was then immersed in silver benzoate to give*Gossypium hirsutum* immersed silver nanoparticle alkyd resin (GHSOR-AgNps)and the formulated resin was analyzed usingUltraviolet-Visible spectroscopy (UV), Fourier Transform Infrared Spectroscopy (FTIR),andScanning Electron Microscopy (SEM). UV-visible spectroscopy was used to analyze the silver benzoate salt. The performance of the formulated resin was assessed and revealed thus: soluble in both polar and non-polar organic solvents used in the study, dried through after 24 hours, showed chemical resistance to 0.1M NaOHwith total removal, and its acid value was below 10mgKOH/g.Therefore,Gossypium*hirsutum* seed oil(GHSO)has been shown to be a cost-effective, renewable and sustainable feedstock for alkyd resin production in the coating industry.

Keywords:Alkyd resin, cotton-seed oil, *Gossypium hirsutum,* physicochemicalproperties, Silver-Nanoparticle,seed oil,Nanoparticles,cotton plant,sodium hydroxide

Introduction

The increased interest in nanotechnology and nanoparticles in particular for more than two decades in various fields of study has necessitated scientists to explore more into the nanoworld. Nanoparticles are tiny or small materialsranging between 1nm to 100nm in diameterandcanbe classifiedinto: one, two or three dimensions (1). Nanoparticles are said to have high surface area to volume ratio,which gives each nanoparticle its unique surface (2). Thedisadvantages of conventional methods in synthesizing nanoparticles includehigh cost, production process time, as well as the use of toxic materials.Recent studieshave employed the use of green, eco-friendly and one-pot synthetic processes,especially in polymer synthesis(3).

The cotton plant is a major crop grown in the world(4). In Nigeria, it is majorly grown in the Northern states and some areas in the South Western states. It is grown mostly for the cotton,

which is used in thetextile industry and for the manufacture of some household materials and industrial products. Cotton seed(*Gossypium hirsutum*) is the second major product from the cotton plant after the cotton fiber which serves as raw material for oil extraction or animal feed production(5,6).Over the years, researchers have usedcotton seed as a renewable raw material in the coating industry; either as a reinforced biomass, used directly as a starting material, or its fibers which are used as a mix for animal feed.(7,8,9,10).

Silver nanoparticles (Ag-Nps) have found use in diverse applications due to their eco-friendly nature (12,13,14).Alkyd resins have been synthesized by various researchers using different methods and catalysts such as: Lithium hydroxide, lead (ll) oxide, calcium carbonate, sodium hydroxide and calcium oxide**(**15,16,17,18,19,20).

This study reports the synthesis of alkyd resin from cottonseed oil, which is eco-friendly,and the immersion of same in silver nanoparticles. The formulated alkyd resin embedded in silver nanoparticleswas characterized using modern analytical techniques such as GasChromatography-Mass Spectrometry(GCMS) for the fatty acid profile of the seed oil extract,Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy(SEM).Ultra–Ultra-violet visible Spectroscopy(UV)was used to characterize the Ag-Nps salt. Theformulated resin wasanalyzedusing standard analytical methodsfor its drying time, chemical resistance, solubility and acid value to evaluate its applicability in the coating industry.

Materials and Methods

Sample preparation

Cotton seed (*Gossypiumhirsutum)* wassourced fromDawanau International Grain Market located at latitude$12^{0}5^{1}$ 12.23^{11}Nand $8^{0}26^{1}27.99^{11}$E longitudeat Dawakin Tofa local government area of Kano stateNigeria. Grits were removed, and theseeds were dehulled, sun-dried, milled into fine powder and stored in an airtight container for further use.

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

Oil Extraction

The *Gossypiumhirsutum*(Cotton seed)oil was extracted via soxhlet extraction.One hundredgrams (100g) of the groundcotton seed was weighed and the oil was extracted using n-hexane (1:2)for 6

hours; the extracted oil was furtherconcentrated using rotary evaporator(RE.300).Weights of the obtained oil extract was noted, and the formula below was used to calculate the % yield.

$$\text{Percentage Yield} = \frac{\text{W2} - \text{W1}}{W2} \; X \; 100$$

W_1= weight of *Gossypium hirsutum*before extraction

W_2 = weight of *Gossypium hirsutum*extracted oil

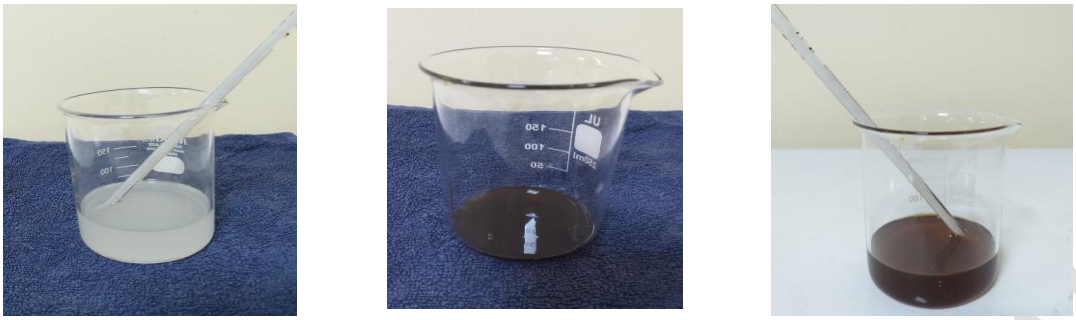
Synthesis of the *Gossypium hirsutum*Seed Oil Extract Alkyd Resin

The resin was prepared using alcoholysis-polyesterification reaction according to the method described by(21,22) withslight modifications.Eighty-five grams(85.0g) equivalent ofthe cotton seed extractwas transferred intoa 4 neck round bottom flaskwhich was heated to a temperature of130^{0}C to debar moisture.Twenty grams (20.0g)volume equivalent of glycerol and a catalytic amount of(0.40g) NaOH was added to the mixture and heated to 220^{0}C under reflux for 1 hour. The reaction was complete when the solubility of the formulated resin in anhydrous methanol gave a clear solution, which gave an indication of the production of monoglyceride.

The reaction temperature was raised to 180^{0}C and phthalic anhydride and 10% v/vxylene were added to the produced resin to distil off the water of esterification. The esterification reaction proceeded at a temperature of 250^{0}C for 4 hours. An aliquot of the reaction mixture was sampled at a time interval of 30 minutes to ascertain a drop in the acid value of the resin. The reaction was stopped when the acid value of the resin dropped to a value below 10 mg/KOH.

Silver Nanoparticle Immersed Alkyd Resin

The procedure for the immersion of the alkyd resin in silver benzoate was the method described by (23) with slight modification. 0.136g of Silver benzoate salt was dissolved in 150ml of toluene.9.60g equivalent of the alkyd resin was dissolved into the mixture and allowed to stand in the dark for a period 12 hours.



(a) (b) (c)

Figure1: showing the solution mixture of (a) silver benzoate salt, (b) freshly prepared alkyd, (c) alkyd resin immersed nanoparticle respectively.

Characterization

Physicochemical properties of the seed oil extract such as; Acid value, saponification value, iodine value, free fatty acid, refractive index and specific gravity analysis were assessed using the Association of Official Analytical Chemists (24) standard analytical method and procedure.

Gas Chromatography Mass Spectra(GC-MS)

The Fatty acid profile of*Gossypium hirsutum* seed oil (GHSO) extractwas carried out. The samples (100 µl) were extracted from the reaction mixture at specified time intervals (7,8 and10 minutes) and analyzed using a Gas Chromatograph, coupled mass spectra (Agilent, 6890A). The GC machine contained a HP-5 capillary column supplied by Agilent with the dimensions 30 m × 0.32 mm× 0.25 µm. The samples were centrifuged for 4 minutes at 50^0C then the top layer of the sample was isolated for GC analysis; with helium as the carrier gas. The column temperature was held at 50^0C for 4 minutes and gradually increased to 300^0C at a heating rate of, 10^0C for 8 minutes.The injector temperature was set at 250^0C while the temperature of the detector was maintained at 300^0C.

Fourier Transform Infrared Spectrometry (FTIR)

Fourier Transform Infrared Spectrometry (FTIR) spectra analysis of the resin produced from*Gossypium hirsutum*seed oil(GHSO) extractand*Gossypium hirsutum* seed oil resin-immersed nanoparticle(GHSOR- AgNp) samples were scanned in the 4000 – 650 cm^{-1} range using CAREY630 machine at 1nm interval.

Scanning Electron Microscopy (SEM)

The morphology of the *Gossypium hirsutum* seed oil extract resin (GHSOR) was examined at different sample sizes of 20um and 50um at a working distance of 3mm and 10.8mm with a

magnification of 9000X and 10000X respectively at a pressure of 70pa at 15KV, and an horizontal field width of 125µm.

UltraViolet-Visible Spectrophotometry

The absorption peak profile for Silver benzoate salt and GHSOR-AgNpwere determined using a UV-visible spectrophotometer at a wavelength range of 200-700 nm.

Evaluation of Formulated Alkyd Resin

The formulated resin from GHSOwas evaluatedfor parameters such as:drying time, chemical resistance, solubility and acid value.

a) Solubility: the resin (0.25g) was weighed and dissolved with 5ml each of acetone, toluene, benzene and methanol(25).
b) Acid value: The acid value of the resin was monitored with the reaction time, which was taken every 30 minutes interval.
c) Drying time: The drying time was evaluated at room temperature, for set to touch,surface dry, dry and dry through.
d) Chemical resistance: This was determined in solutions such as:distilled water,0.1M HCl, 0.1M NaOH and 5% NaCl respectively.

Results and Discussion

Extraction yield

The yield of *Gossypium hirsutum*seed oil (GHSO) extract was achievedas 26.67%This yield varies from one cotton seed plant to anotheras a result of temperature, planting season, storage and extraction process among other factors(26).

Physicochemical Properties

The physicochemical properties of *Gossypium hirsutum* seed oil extract are shown in Table 1. The acid value and free fatty acid of the seed oil extract of *Gossypium hirsutum* was 3.19 mgKOH/g and 1.60 mgKOH/g respectively. The acid value is used to determine the deterioration and curing of the oil, and the value obtained was similar to that reported by (27).

As shown in Table 1,Iodine value is one of the most important parameters to look out whenconsidering oil for resin preparation. Iodine value is used to determine the drying property of an oil (28).

Iodine value classifies oils as non-drying; when the value is less than 90 $gI_2/100g$, semi-drying when it value falls between 90-130$gI_2/100g$, and drying when it is higher than 130$gI_2/100g$.The iodine value for *Gossypium hirsutum*was found tobe 102.36 $gI_2/100g$ which indicates that it is a semi-drying oil, this is similar to that reported by(29)

The specific gravity of *Gossypium hirsutum*seed oil was found to be 0.91 g/cm^3. This value is similar to0.93 g/cm^3reported for rubber seed oil and cotton seed oilby(30,29) respectively;and also within the range reported by(31); value for sunflowerseed oil.

The refractive index(RI)can be used to test for oil purity. TheRI value for GHSO was foundto be 1.46;this value falls in the (RI) range reported by(30, 21)for most seed oils.

The hydrocarbon carbon (HC) chain can be determined by saponification value (SV), The higher the hydrocarbon chain the lower the saponification value. The saponification value forGHSO is 202.32mg/KOH/g which is in the same range as that reported by(29,32) Thecolour of GHSO was physically observed as golden yellow, the odour is agreeable and the moisture content was found to be 1.20%which aligns with observations reported by(9)

Table 1:Physicochemical Parameters of *Gossypium hirsutum*Seed Oil Extract

S/N	PARAMETERS	GHSO
1	Acid value (mgKOH/g)	3.19
2	Free fatty acid	1.60
3	Iodine value ($gI_2/100g$)	102.36
4	Specific gravity g/cm^3(30^oC)	0.91
5	Refractive index	1.46
6	Saponification value (mg/KOH/g)	202.32
7	Moisture content (%)	1.20
8	Viscosity(30^0C) (m^2/S)	42.00
9	Percentage yield (%)	26.67
10	Odour	Agreeable
11	Colour	Golden yellow

The fatty acid composition of *Gossypium hirsutum*seed oil extract is as Shown in Figure 2. The most abundant unsaturated fatty acid is oleic acid(7.45ppm) (octadec-9-enoic acid) and the least is linoleic acid (octadec-9, 12-dienoic acid)(0.02ppm) this is reported by(29). The most abundant saturated fatty acid is heptadecanoic acid(4.24ppm) (Margaric acid) and the least is Arachidonic

acid (cis-9-cis-12-cis-15-cis-19-Eicosanoic acid)(0.09ppm). The total saturated and unsaturated fatty acid of *Gossypium hirsutum* is 26.62% and 75.72% respectively which compares favourably with most literatureon cottonseed oils(33,29).The iodine value obtained for GHSO 102.36 gI_2/100g is confirmed by the saturated and unsaturated fatty acid profile of the seed oil. which confirms it use as a viable feedstock for resin production.

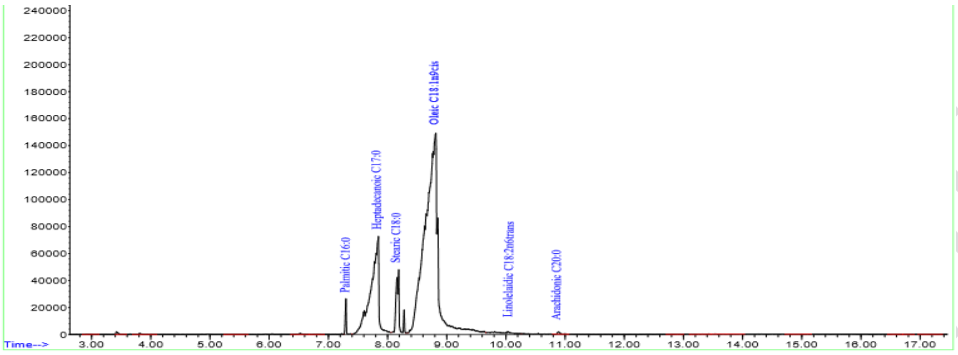


Figure 2: GCMS fatty acid profile of *Gossypium hirsutum*seed oil (GHSO) extract

The FTIR spectra of the alkyd resin seed oil formulated from GHSOR and GHSOR-AgNpsare shown in Figures 3 and 4 respectively.Similar functional groups which correspond to C-H aliphatic stretch absorption bands were observed at 2925cm-^{1}and2825 cm-^{1} for both GHSOR andGHSOR-AgNps. Olefinic C-H stretching vibration peaks were observed at 3011 cm-^{1} and 3089 cm-^{1}respectively for bothresins. Absorptionpeaks at 1748 cm-^{1} and 1741 cm-^{1} arise from conjugated carboxylicC=O ester bonds.The observed peaks at 1465 cm-^{1} and 1499 cm-^{1} correspond to C=C stretches arising from symmetric and asymmetric bending of the methyl groups. The absorption band observed at 1607 cm-^{1} is due to C=C aromatic stretching vibration which confirms that thepolyesterification reaction was successful.The newabsorption peak at 698 cm-^{1}is due to Ag-O bond indicative of the formation ofGHSOR-AgNpsas this peak is non-existence in the GHSOR (figure 3), same was observed by(34,20,22).

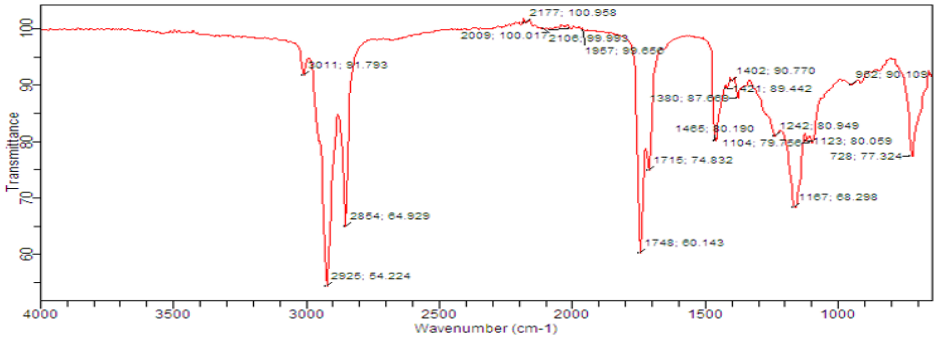


Figure 3: FTIR of Alkyd resin from *Gossypium hirsutum*seed oil extract (GHSOR)

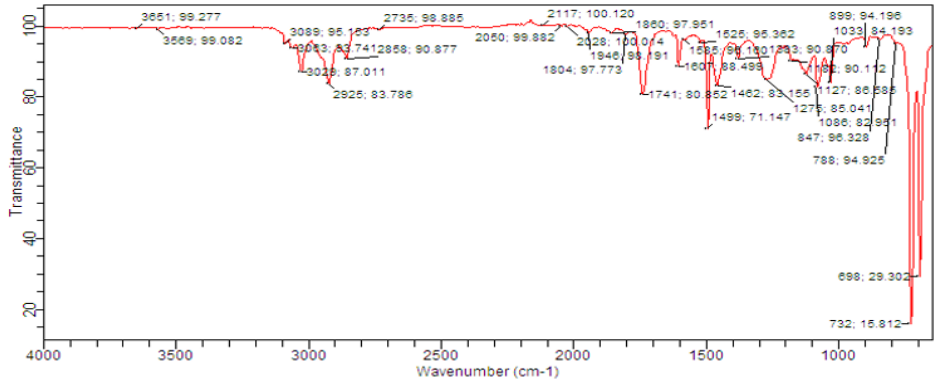
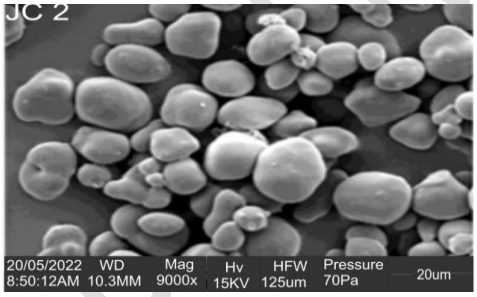
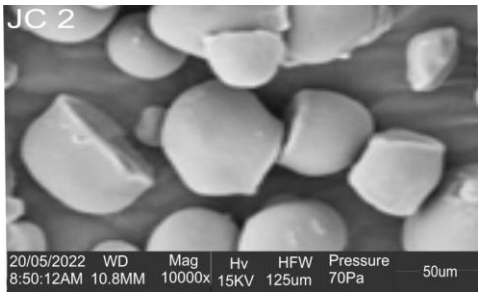


Figure 4: FTIR of silver nanoparticle immersed on *Gossypium hirsutum*seed oil alkyd resin (GHSOR-AgNp)

On examination of the SEM micrograph shown inFigure 5, the morphology of the resin at 9000x and 10000x resolutions shows they were spherical in shape with good and uniform roughness and dispersion.



(a)



(b)

Figure 5: SEM Micrograph of GHSOR showing (a) 20µm (b) 50µm

The UV-Visibleanalysis of silver benzoate salt and GHSOR-AgNpsis shown in Figure 6.The maximum absorption wavelength was seen at 283nm for Ag^{+} ion and 459nm for Ag-Nps due

tosurface plasmon resonance (SPR),which is a confirmation of the resonance effect due to the interaction of conduction electrons of metal nanoparticles (22).

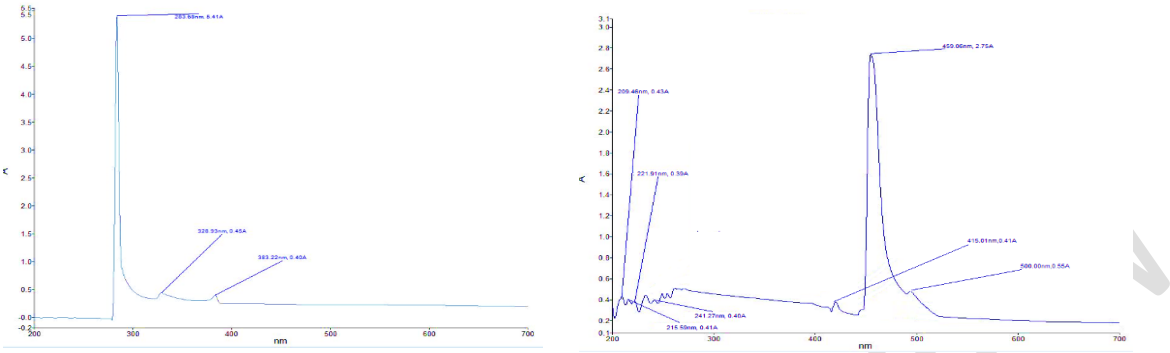


Figure 6: UV-visible spectra of (a) silver benzoate salt, and (b) alkyd resin immersed nanoparticle

Performance Evaluation of the Prepared Resin

The formulated resin (GHSOR) was evaluated for its solubility, drying time and chemical resistance as shown in tables 2, 3, and 4 respectively.

The resin was found to be soluble in acetone, toluene and benzene which are polar and non-polar solvents and partially soluble in methanol. Asimilar observation was reported by(35).The drying time of theresin was assessed at different time intervals of 30 minutes to 24 hours. At 30 minutes, the resin was set to touch, which is a touch on the surface with little smear on the hand; surface drying was observed at 1 hour timing and drying at 3 hours which is convenient to touch.The dry-through was observed after 24 hoursthis shows a property comparable to other literature reported for oil-modified alkyd resins (9,25).

Table 2: Solubility of the formulated resin

Acetone	**Toluene**	**Benzene**	**Methanol**
soluble	soluble	soluble	Partially soluble

Table 3: Drying time of the formulated resin

Appearance	**Set to touch**	**Surface dry**	**Dry**	**Dry through**
drying time schedules	30minutes	1 hour	3 hours	24 hours

The chemical resistance of the resin was assessed after 24 hours.No effect was observed on the alkyd resin immersed in distilled water at0.1M NaCl and 0.1M HClwhich was sufficient time to test for resistance. At 24hours there was removal on 0.1M NaOH which shows that the resin formulated has a high chemical resistance to alkali solvents, this was also reported in literature(36,37).

Table 4: Chemical resistance of the formulated resin

Chemicals	Observation	Immersion time
Distilled water	No effect	24 hours
0.1M HCl	No effect	24 hours
0.1M NaOH	Removal	24 hours
5% NaCl	No effect	24 hours

The acid value of the formulated resin (GHSOR) was determined at different reaction time. A plot of these values at different reaction time intervals is illustrated in Figure 7. The plot showed a sudden drop in the acid value atthe first 30minutes then a gradual drop of the acid value to a value below 10mgKOH/gwhich is due to the reaction of 1^{o} and 2^{o} –OH groups of glycerol with the -COOHgroups of phthalic anhydride. This is similar to that reported in the literature for most seed oils (36,27).

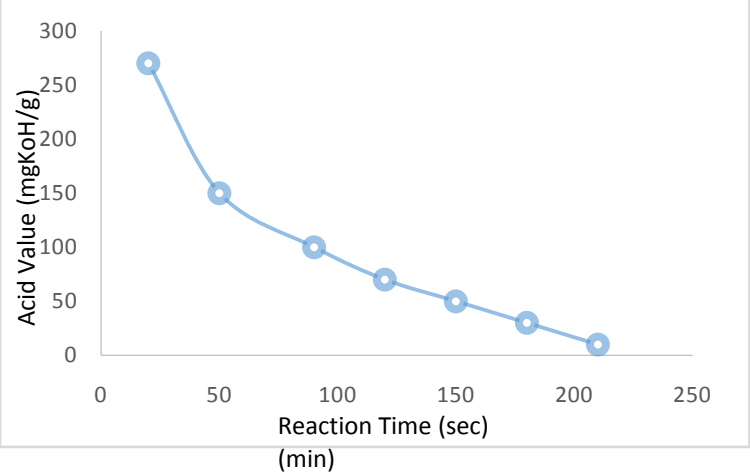


Figure 7: Acid value versus reaction time of GHSOR

Conclusion

From this study, it can be concluded that alkyd resin based on pure*Gossypium hirsutum* (GHSO) has been successfully synthesized and immersed in silver nanoparticles. The FTIR confirms the chemical structures of the resin and the functional group of the resin immersed in silver nanoparticles (GHSOR-AgNps); hence *Gossypium hirsutum*is a good starting material in the coating industry.

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