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SYNTHESIS, CHARACTERIZATION AND APPLICATION OF SILVER-NANOPARTICLES-EMBEDDED ALKYD RESINS DERIVED FROM JATROPHA SEED OIL

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ABSTRACT

This research was carried out to synthesise, characterise and evaluate the performance properties of silver-nanoparticles embedded alkyd resin as binder in surface coating. Locally available Jatropha Seed Oil (JSO) was successfully converted to alkyd resin by alcoholysis and polycondensation reactions. The oil was reacted with glycerol to form monoglyceride which react further with phthalic anhydride to form the alkyd resin (JSOR) as monitored titrimetrically. Silver nanoparticles (AgNPs) were also synthesized from silver benzoate solution (SBS) by *in situ* reduction method. The SBS was mixed with JSOR in toluene to form a homogeneous mixture which subsequently formed silver nanoparticles-embedded alkyd resin (AgNPs-JSOR). The formation of AgNPs-JSOR was confirmed by colour change, UV-visible spectrophotometry which showed a surface plasmon resonance at 455 nm and FTIR spectroscopy which revealed a stretching vibration of Ag-O bond at 696 cm^{-1}. The antimicrobial activity of SBS, JSO, JSOR, and AgNPs-JSOR were evaluated against different pathogenic bacteria and fungi: *Escherichia coli, Salmonella typhi, Staphylococcus aureus Aspergillus flavus, Aspergillus fumigatus and Mucor species* using the disc diffusion method. The appearance of higher zone of inhibition (20-25 mm) in AgNPs-JSOR clearly indicated a higher antibacterial and antifungal activity when compared to SBS, JSO and JSOR. The performance characteristics of the JSOR and Ag-NPs-embedded alkyd resins as ingredients in surface coatings (paint) were further evaluated. The results obtained revealed that the paint films air-dried within few hours, showed good solvent resistance and excellent light fastness suggesting the potentiality of AgNP-JSOR as binder in antimicrobial surface coatings.

Keywords: Jatropha seed oil, Alkyd resin, Silver nanoparticles, Plasmon resonance, FTIR, Antimicrobial

INTRODUCTION

The National Nanotechnology Initiative (NNI), United States of America (USA) defined Nanoparticles as microscopic particles with at least one of the three dimensions less than 100 nanometre (nm) (Behera *et al.,* 2013). In recent years, nanoparticles have received enormous attention for the creation and manipulation of products at Nano scale level having novel properties (Hussain *et al.,* 2005). The properties of nanoparticles entirely differ from conventional macroscopic materials. These unique characteristic properties of nanoparticles arise from the high surface to volume ratio (Premanathan *et al.,* 2011). AgNPs are water dispersible nanometre sized, that can be produce by chemical reduction using a reducing agent such as

$$AgNO_3 + NaBH_4 \longrightarrow Ag + \tfrac{1}{2}H_2 + \tfrac{1}{2}B_2H_6 + NaNO_3$$

Ag-NPs have been in use for more than 150 years and are recognized as antimicrobial agents in United States (USA) since 1954 (Adnan and Kang, 2014). Silver nanoparticles (Ag-NPs) are world widely famous for their versatility which they exhibit in terms of applications such as electrical, thermal, and antimicrobial properties among others. In the past people used Ag for ornamental, medicinal, crockery, clothing, building materials, and coins making and as disinfectant. Reports suggested that Ag-NPs have no side effect on the body and act as antimicrobial agents when used with proper care (Panyala *et al.,* 2008). Previously silver salts were used for the treatment of mental disorders, drug addiction, and several infectious diseases caused by pathogens. The most stable oxidation state of Ag is 0 and +1 although it can exist in other oxidation states as well and can form various complexes. The size and geometry of Ag-NPs are dependent on the synthetic route adopted for its synthesis; however it can be found in spherical, rod, and triangular shape, or coated with polymer, biomolecules, and sugars (Adnan and Kang, 2014).

Variety of methods, such as chemical, physical, photochemical, and biological, has been employed for the synthesis and stabilizing of Ag-NPs. Each method has its merits and demerits. The synthetic routes for Ag-NPs are commonly associated with problems such as costs, stability, scalability, particle sizes and size distribution. Among the existed reported methods, so far, chemical methods are preferred for the preparation of Ag-NPs due to the ease in synthesizing them in solution. Many researchers are using

these methods to synthesize Ag-NPs in various sizes and shapes. For example, one research group synthesized monodispersed silver nanocubes by simply reducing $AgNO_3$ with ethylene glycol in the presence of polyvinylpyrrolidone (PVP) polymer (Sun and Xia, 2002). The process was called polyol process. In this process, it has been revealed that ethylene glycol works as both the solvent and the reducing agent. Furthermore, the size and shape of the nanocubes were dependent on the molar ratio of $AgNO_3$ and PVP. Thus by controlling the experimental parameters, the geometry (size and shapes) of the Ag-NPs can be tailored. Round shaped Ag-NPs with a controlled size and monodispersity were synthesized by modifying the polyol method using precursor injection. In this method, particles with 20 nm or smaller size were prepared. The governing factors of the precursor injection method were precursor injection rate and *in situ* conditions. The injection precursor method proved to be effective in synthesizing particles with brilliant control on the size for the monodispersion (Kim *et al.,* 2006). Normally, the synthesis of Ag-NPs by chemical method banks on type of Ag precursor, reducing agents, and stabilizing agents. Furthermore, the synthesis and geometry of Ag-NPs rely on the nucleation and subsequent stacking of the Ag nuclei. Uniform size and monodispersity can be achieved by controlling the nucleation stage and stacking of nuclei in which intern depends on experimental parameters such as precursor, pH, temperature, and reducing agents (Kim *et al.,* 2006; Kumar *et al*., 2008; Adnan and Kang, 2014).

In this work jatropha-based alkyd resin is used in the synthesis and stabilition of AgNPs without the use of external reducing agent but an appropriate choice of the organometallic salts, for example, silver benzoate, facilitates the solubility of nanoparticle precursors into the oil medium. Hence, it is anticipated that silver salts undergo ligand exchange with fatty acids, which causes the metal ions to dissolve in the oil and subsequent reduction by the free radicals to form nanoparticles. This method is an environmentally friendly procedure and more favourable in view of it's low cost of production and minimum time required as compared to chemical reduction methods using a variety of organic and inorganic reducing agents.

MATERIALS AND METHODS

Materials

Silver benzoate salt (SBS) was purchased from Sigma Aldrich and used as received. Jatropha seed oil (JSO) was purchased from Federal Technology Incubation Center, Kano (FTICK). All chemicals and solvents used were of analytical grade and used without further purification. The glasswares used were washed thoroughly, rinsed with de-ionised water and dried in oven overnight before used. All weighing were carried out on an electric Mettler Balance Model H30AR. FTIR Spectral Analysis of the jatropha seed oil, alkyd resins and silver nanoparticles samples were recorded on FTIR CARY 630 Agilent Technologies in the range of 4000 – 650 cm^{-1} in the Instrumental Laboratories, Department of Pure and Industrial Chemistry, Bayero University, Kano. Viscosity measurements of alkyd resin, formulation and performance evaluation of paint samples were carried out in the Polymer Research laboratory in the Department of Pure and Industrial Chemistry, Bayero University, Kano. Antibacterial and Antifungal activity studies were carried out at the Research Laboratory of the Department of Microbiology, Bayero University, Kano.

Methods

Decolorizing of Jatropha Seed Oil (JSO)

100g of Jatropha seed oil was mixed with 10g of activated charcoal and stirred for 30minutes and then heated to 100°C for 1hour, the mixture was cooled to room temperature and activated charcoal was removed by filtration (Hlaing *et al.,* 2008).

Physicochemical Characterisation of JSO

The pysicochemical properties of JSO such as refractive index, iodine value, saponification number, acid value, percentage free fatty acid and specific gravity were determined according to standard methods (AOCS, 1996).

Synthesis of Jatropha Seed Oil Alkyd Resin (JSOR)

The synthesis was carried out according to the procedures reported by; (Onukwli *et al*., 2008 and Oladipo *et al.*, 2013). Jatropha seed oil (80.0g) was poured into the three necked flask. This mixture was heated on a heating mantle to about 120ºC to expel moisture and 20.0g of glycerol was added at this temperature before raising the temperature to 230ºC. After 40 minutes, a small quantity of the reaction mixture (aliquot) was taken to check for solubility in methanol, which gave a clear solution that indicated the formation of the monoglyceride at the end of the first stage. At the beginning of the second stage, the temperature was lowered to about 180ºC and 46.0g of phthalic anhydride was added, followed by addition of about 10% xylene into the reaction mixture to aid distilling off water of esterification by forming an azeotrope. The temperature was gradually increased to 240 – 250ºC and maintained for about 4 hours. Aliquots were withdrawn from the reaction mixture at intervals of thirty minutes to check for drop in acid value and the reaction was stopped when the acid value was below 10 mg KOH g-ml to allow the cooling of the alkyd resin.

Physicochemical characterisation of Jatropha Seed Oil Alkyd Resin (JSOR)

The physicochemical properties of the alkyd resin prepared from the jatropha seed oil; such as acid drops values using American Oil Chemist's Society methods (AOCS, 1996).

Synthesis of Silver Nanoparticles embedded Alkyd Resin (AgNPs - JSOR)

The synthesis was carried out according to the procedure reported by (Kumar *et al*., 2008). Silver benzoate (0.068g) was dissolved in 75 mL of toluene. Alkyd resin (4.8 g) was mixed to form a homogeneous solution and kept in dark for 12 hours. This is as shown in plate 1.

0.068g of Silver benzoate salts was dissolved in 75ml of toluene

4.8g of Alkyd Resins

Homogeneous mixture of Silver benzoate salt and Alkyd Resins

Plate 1: Synthetic route of silver nanoparticle (AgNPs

Characterisation of JSOR and AgNP-JSOR

FTIR of JSOR and AgNPs-JSOR

The FTIR spectra of CSO and AgNPs-CSOR were acquired using FTIR spectrometre CARY 630 Agilent Technologies. The spectra were recorded in the frequency range of 4000-600 cm^{-1} at 1 nm interval to identify the absorption peaks.

UV-Visible Spectrophotometry of Silver Benzoate Salt (SBS) and Silver Nanoparticles embedded Alkyd Resin (AgNPs-CSOR)

The UV-visible absorption profile of silver benzoate salt (SBS) and silver nanoparticles embedded alky resin samples (AgNPs-CSOR) were determined using UV-visible spectrophotometre at wavelength range of 200-750 nm.

Antibacterial and Antifungal Activity of SBS, JSO, JSOR, JSOR-AgNPs

Disc diffusion method was used, in which the agar plates were incubated with test organisms: *E. coli*, *Salmonella typhi*, *staphylococcus aureus* and *Aspergillus flavus*, *Aspergillus fumigatus and Mucor species* respectively *by* spreading uniformly. One disc from each sample was placed in the petri-discs with sterile forceps. The discs were incubated for 24 hours at 37^{o}C. After 24 hours, the antibacterial and antifungal activity was determined by measuring the zone of inhibition and the value were compared with standard drugs (Ampicillin and Ketoconazole) the results are presented in Figure 5.

Formulation of Paint Using Synthesized Binder

Different paint formulations were prepared by measuring 10g of alkyd resin derived from jatropha seed oil and silver nanoparticle-embedded alkyd resin. In each formulation, iron (II) oxide was mixed with solvents before adding to the alkyd resins. Also paint additives such as extenders and thickness were added and stirred to obtain a homogeneous mixture.

Table 1: Composition of Surface Coating (Paint) Formulation

Components	**Weight in grams**		
	Blank	**JSO**	**AgNPs-JSOR**
Alkyd Resin	0.0	10.0	10.0
Iron (ii) oxide	7.50	7.50	7.5
Xylene	30	30.0	0.0
Talc	0.8	0.8	0.8
Nitrosol 0.3	0.3	0.3	
Pigment Volume Concentration (PVC) 0.0	0.085	0.085	
%PVC	0.0	8.5	8.5

Characteristics of Formulated Paint Films

The performance characteristics of the films were determined in terms of chemical resistance, drying schedules, and light fastness. Films of alkyd resins derived from jatropha seed oil and silver nanoparticle-embedded alkyd resin drived from jatropha seed oil paint was prepared by applying a thin spread of the resin on clear glass panel and dried at room temperature. The drying processes were monitored in terms of the time of set-to-touch, surface-dry and dry-through. The chemical resistance was determined using ASTM (D 1308 – 67). Standard test method was used to evaluate the resistance of the paint films to different solvent media (H_2O, KOH, H_2SO_4, and NaCl) at room temperature.

RESULTS AND DISCUSSION

Physicochemical Characterisation of Jatropha Seed Oil (JSO)

The results presented in Table 2 show that JSO is light yellow in colour after decolorization with activated charcoal. The acid value of the JSO was found to be 9.24. The acid value is the measure of the extent to which the continents glycerides have been decomposed by lipase actions. The iodine value of JSO was found to be 62.40, which is less than 100, hence the JSO is classified as non-drying oils. Iodine value measures the degree of unsaturation of oil. The iodine value is lower than 100, shows that the JSO has lower degree of unsaturation. Also, the higher the iodine value, the higher the degree of unsaturation and the more tendency the oil will have to undergo oxidative rancidity (Bora *et al.,* 2014). The saponification value of JSO was found to be 196.5. This indicates the average molecular mass of fatty acids present in oil. The peroxide value of JSO was found to be 36.10meq/kg. This indicates the non- rancid nature of the oil. The percentage free fatty acid for JSO is 4.8% which can be associated to the presence of impurities in crude JSO that could cause hydrolysis of the ester linkages, thereby increasing the free fatty acid level (Nkafamiya *et al.,* 2010; Haruna and Sharif, 2016).

However, the refractive index of JSO is 1.47. It shows the values fell within the range of non-edibility oils. The specific gravity for JSO is 0.9131 which implies that they are less dense than water (Momodu *et al.,* 2011). Also, the percentage moisture content of JSO is 0.59%.

Table 2: Physico-chemical characterisation of JSO

Property	Value
Colour	Light yellow
Acid value	9.24
Iodine value	62.40
Saponification value (mg/g)	196.5
FFA (%)	4.18
Refractive index	1.47
Specific gravity g/cm^3@ 25^{o}C	0.9131
Peroxide value Meq/kg	36.10
Viscosity MPa @ 28^{o}C	91.53
Moisture content (%)	0.59

Titrimetric Determination of Acid Value

Fig.1 shows decrease in acid value with reaction time. It was observed that the acid value initially decreased, this was followed by a more gradual decrease. The decrease in acid value arose from the reactivity of primary and secondary hydroxyl groups of glycerol with carboxyl groups of phthalic anhydride. At the initial stage primary hydroxyl groups of the monoglyceride reacts more rapidly than the secondary hydroxyl groups with carboxyl groups of phthalic anhydride. It is recognized at a temperature of about 160 ^{o}C, while secondary hydroxyl groups reacted at temperature above 230 ^{o}C. Hence, the initial noticeable decrease in acid value perceived for the alkyd resin (JSOR) due to increase in the rate of esterification associated the rapid increase in temperature to 230^{o}C Similar results were reported by (Ikhuoria et al., 2007 and Oladipo *et al.*, 2013)

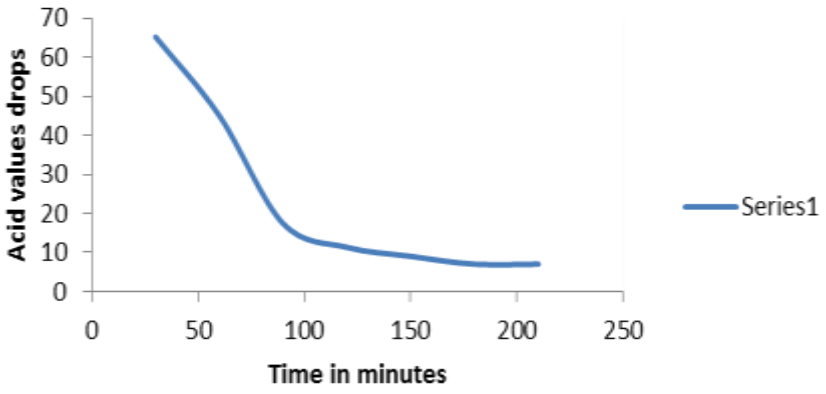

Fig. 1: Plots of drop in acid value vs reaction time of JSOR

FTIR Analysis

Fig. 2 and 3 represent the FT-IR spectra of JSOR and JSOR-AgNPs. The absorption bands at 1743 and 1791cm^{-1} are as a result of conjugation of the aromatic ester due to C=O, bands at 1259 and 1259 cm^{-1} are due to conjugation of the aromatic ester to C–O. The band which appears at 1603cm^{-1} is due to C=C stretching vibration of aromatics. The presence of these peaks has confirmed that polyesterification reaction has taken place. Similarly, on the comparison of the FT-IR spectra of JSOR and JSOR-AgNPs. A new peak appears at 696cm^{-1} which is ascribed to the presence of Ag-O bond. Hence, suggests the successful preparation of JSOR-AgNPs. In the case of synthesized alkyd resins (JSOR), this peak due to Ag-O bond vibration was absent. Similar results were reported by Patel, (2007); Hlaing and Oo, (2008); Guran *et al.,* (2012); Bora *et al.,* (2014)

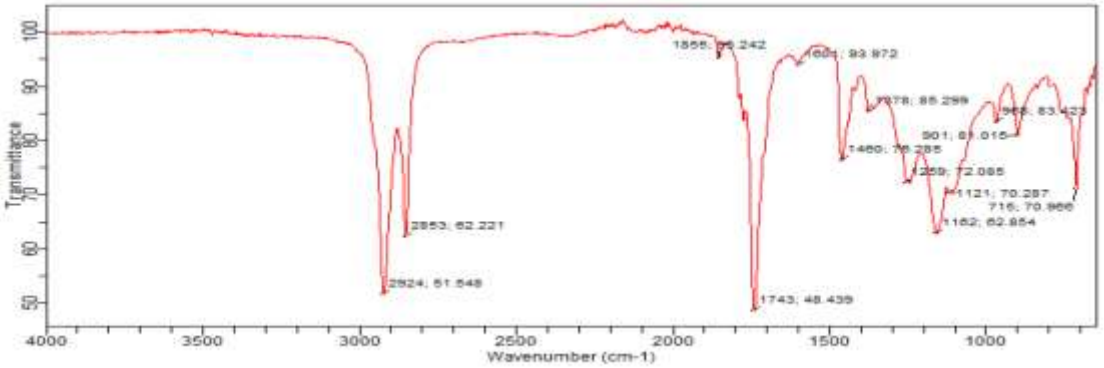

Fig 2: FTIR of Alkyd resin derived from jatropha seed oil

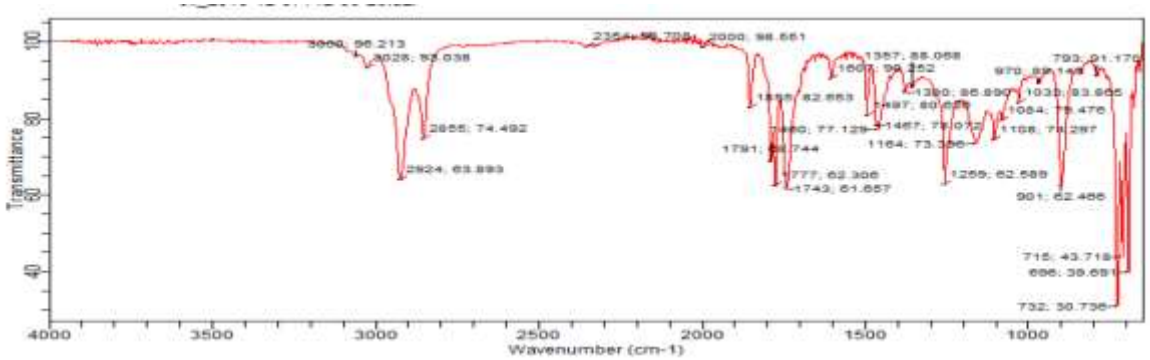

Fig 3: FTIR of Silver nanoparticle-embedded on jatropha seed oil modified alkyd resins

UV-visible Analysis

Fig. 4 shows the results of wavelength of maximum absorption of the SBS and JSOR-AgNPs formed in the reaction media. SBS solution has wavelength of maximum Absorption of 205nm as expected for Ag+ ions and JSOR-AgNPs becomes light yellow on the solution phase as shown in plate 3.2 after 12 hours due to formation of silver nanoparticles. The result of absorbance measurements for JSOR-AgNPs colloidal solution shows an absorption maximum at 455nm which indicated the presence of silver nanoparticles. This peak appears for the Ag-NPs owing to the surface plasmon resonance effect originating from the quantum size of the Ag-NPs, has distintic light yellow colour on the solution phase which did not appear on the homogenous mixture as shown in plate 3.1 that again confirms the formation of silver particles at nanoscale dimensions. The absorbance maximum does not change over a long period, indicating that the silver particles are prevented from coagulating owing to stabilization of nanoparticles by fatty acids, which are essential constituents of the JSO. Similar results reported by Dhand *et al*., (2015).

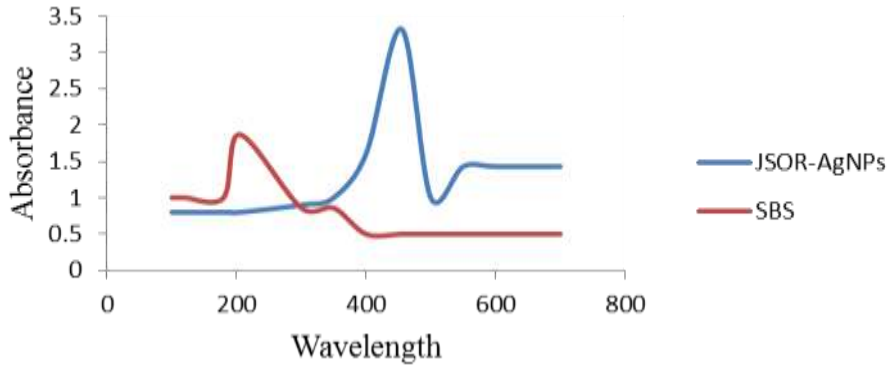

Fig. 4: shows the results of wavelength (nm) of maximum againts Absorbance of SBS and JSOR-AgNPs

Plate 2: Homogeneous mixture of Silver benzoate salt and Alkyd Resins

Plate 3: This picture show light yellow colour on the solution phase due the formation of AgNPs

Antibacterial and Antifungal Activity Evaluation by Disc Diffusion Method

Fig. 5 shows the antibacterial and antifungal activities test for the SBS, JSO, JSOR and JSOR-AgNPs.The diameter of Zones of inhibition was measured (mm) for each treatment, antibacterial and antifungal effect were studied against three different pathogenic bacteria; *E.coli*, *Salmonella typhi and Staphylococcus aureus* and three different pathogenic fungi namely; *Aspergillus flavus*, *Aspergillus fumigatus* and *Mucor specie*. Solution dispersion SBS, JSO, JSOR, JSOR and JSOR-AgNPs of desired concentrations were prepared. The concentrations were measured in µg/ml. It is clearly shown that JSOR-AgNPs had the higher antibacterial and antifungal activities compared to SBS, JSO and JSOR. Similar results reported by (Shaker *et al*., 2012, Rivero *et al.*, 2011 and Cornelia *et al*., 2012)

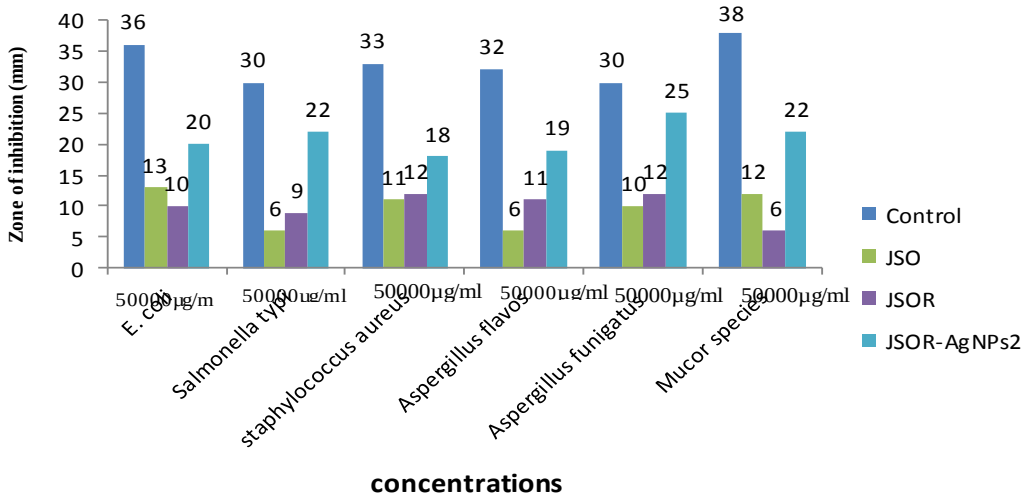

Fig. 5: Antibacterial and antifungal activity of JSO, JSOR and JSOR-AgNPs2

Performance Evaluation of the Prepared Paint Films

Table 1 shows the various components formulations of alkyd resins derived from jatropha seed oil and silver-nanoparticles-embedded alkyd resins paints. From the table, two different formulations containing JSOR, JSOR-AgNPs are formulated with toluene and pigment-volume-concentration (%PVC) is 4.0. PVC is an important parameter that can be used to determine the quality of paints. The lower the PVC, the higher the durability and glossy of paint. Also, with increase in PVC, adhesion as well as durability decrease. If the volume increases as compared to the volume of binder, the film will lose cohesion. The paint will be in powdered form and obviously will have little durability. The blank sample has little durability owing to the absence of polymeric binder.

Drying Schedule Analysis

Fig 6 shows the drying schedule of the different formulated paint JSOR, and JSOR-AgNPs respectively. The results show all blank samples have very fast drying property owing to the absences of binder. JSOR-AgNPs showed an excellent dying property on comparison with JSOR.

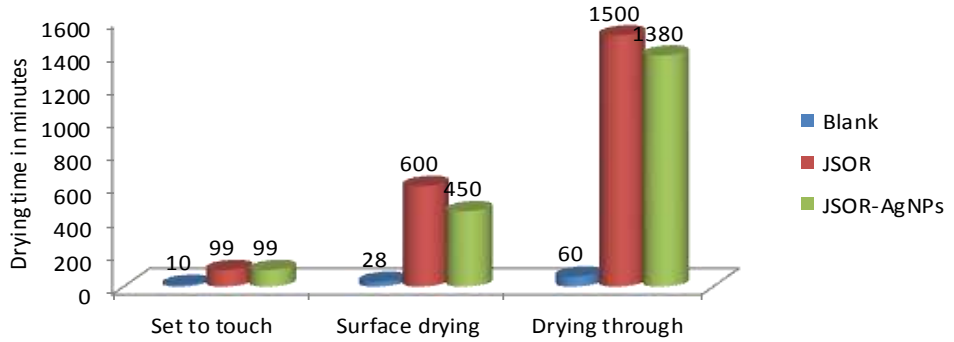

Fig. 6: Drying schedule of the different formulated paint JSOR and JSOR-AgNPs2

Chemical Resistance Test

Table 3 shows the chemical resistance characteristics of blank, JSOR, JSOR-AgNPs respectively. From the results obtained, it shows that blank film have poor resistance to acid, brine and alkali whereas JSOR and JSOR-AgNPs films show an excellent resistance to acid, brine and water but poor resistance to alkali because of presence of an ester group in the binder.

Table 3: Chemical Resistance of the Formulated Paints JSOR and JSOR-AgNPs

Test	Blank	JSOR	JSOR-AgNPs
Alkali (0.1M KOH)	2	2	2
Acid (0.1M H_2SO_4)	2	1	1
Brine (5% w/w)	2	1	1
Distilled water	1	1	1

Key: 1 = Film not removed
2 = Film removed

Light Fastness Rating

Table 4: show the consequences of light fastness test, of JSOR and JSOR-AgNPs, respectively. Using artificial light for 120 hours, from the results, JSOR, and JSOR-AgNPs shows an excellent light fastness property while blank sample shows moderate light fastness property due to the absence of binder in the blank sample.

Table 4: The light fastness test of the formulated paints

Sample Grade	Degree of fading	Light fastness type
Blank	4	Appreciable fading Moderate
JSOR	7	Very slight fading Excellent
JSOR-AgNPs2 7	Very slight fading	Excellent

CONCLUSION

This research shows that the locally available *Jatropha curcas* seed oil has been successfully used in the synthesis of alkyd resins by Alcoholysis and Polycondensation. Silver benzoate salts have been successfully converted to silver nanoparticles (AgNPs) by environmentally friendly *in situ* reduction method. The silver nanoparticles embedded alkyd resins (AgNPs-JSOR) derived from jatropha seed oil showed good antibacterial and antifungal activity. Performance evaluation of the resin shows that AgNPs–embedded alkyd resins have excellent coating properties such as drying schedule, solvent/chemical resistance and light fastness property, hence AgNPs-JSOR would have a promising application as binder in surface coatings with excellent antibacterial and antifungal properties.

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