

Drying and adhesive properties of novel surface coatings derived from peanut skin extract and cashew nutshell liquid

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Abstract

Purpose – This study aims to synthesize pigment and resin from agro-wastes and use them in the formulation of eco-friendly surface coatings.

Design/methodology/approach – The pigments and resin were synthesized through a chemical modification of agro-wastes. The pigments were characterized by infrared spectroscopy (FTIR) and were screened for their antimicrobial activities. The physicochemical characteristics of the cashew nutshell liquid (CNSL)-modified resin were evaluated. These precursors and other natural additives were used to formulate surface coatings, and their drying and adhesive properties were evaluated using international testing methods.

Findings – It was observed that the curing of the CNSL-modified resin depended on time and temperature. The pigments exhibited antimicrobial activity against *E. coli* and *S. aureus* and had high melting points, affirming their stability. The chemically modified precursors successfully yielded surface coatings with acceptable drying times and adhesion to the base substrate.

Practical implications – The use of agro-wastes as the main components of the surface coatings implies waste valorization, a reduction in production costs and the creation of job opportunities for sustainable development. To increase the chemical, physical, corrosion resistance and antimicrobial qualities of paint compositions, chemically modified peanut skin extracts and CNSL can be used as pigments and resins, respectively. This could be a green approach to achieving the targets of Sustainable development goals 11 and 12.

Originality/value – The paper outlines a prospective approach to use unwanted waste (peanut skin, cashew nutshells) and other natural additives as industrial raw materials. These novel surface coating precursors are cost-effective, readily available, eco-friendly and could replace conventional precursors.

Keywords Peanut skin, Cashew nutshell liquid, Pigment, Resin, Surface coatings, Drying time, Adhesion

Paper type Research paper

1. Introduction

Coatings are liquefiable polymeric products that are applied to the surfaces of objects to perform decorative and protective functions. Surface coatings are made of pigments, binders (resins), solvents and additives (Kyei *et al.*, 2020a, 2020b). The pigments perform the coloring function, while the binders grip the pigments together to generate a dry film on the substrate. In most cases, the application of coatings is aimed at performing a protective function by combating fouling (Yong *et al.*, 2019),

microorganisms (Jana *et al.*, 2019), corrosion (Taylor, 2001), mechanical wear (Lima *et al.*, 2013) and environmental change (Kubba, 2010). Coating materials have varied uses in many enterprises in the construction and plumbing sector (Karak, 2016; Wei *et al.*, 2020), electronic and semiconductor industries (Licari, 2003), marine industries (Li and Ning, 2019), aerospace and automotive industries (Ramon *et al.*, 2018), medical appliances and daily domestic applications (Mahadik *et al.*, 2016).

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Although conventional coatings enhance the surface protection of substrates, they have undesirable environmental effects because of the toxicity, carcinogenicity and biocidal activities of the additives used in the formulations. Remarkably, coating ingredients such as tributyltin (TBT), potentially toxic metals such as copper (Cu), cobalt (Co), cadmium (Cd) and volatile organic compounds are not eco-friendly and could cause human health hazards (Garcia *et al.*, 2015; Nine *et al.*, 2015). After several studies confirmed its toxicity even at low concentrations, the once-efficient TBT was banned (Jayeola *et al.*, 2018; Peres *et al.*, 2015) for use as an ingredient in surface coatings. In a recent study undertaken by Rossini *et al.* (2019), the biotoxicity and life cycle of two commercial antifoul coatings were assessed. They found that applying these coatings on ship hulls resulted in the release of copper, zinc, volatile organic compounds and heavy metals into the aquatic environment. This development resulted in the deaths of some aquatic species, which is a threat to the ecosystem (Rossini *et al.*, 2019). Hence, a strategic objective in modern coating manufacturing is to find and use eco-friendly alternatives to replace the toxic coating components. There is, therefore, the need to advance the search for non-polluting and eco-friendly surface coatings, especially from natural raw material sources.

In recent times, several studies have considered the application of natural additives or modifying agents rather than commercial biocides in the formulation of coatings (El-Bindary *et al.*, 2019; Karak, 2016; Noor *et al.*, 2015; Sánchez-Lozano *et al.*, 2019). Natural additives, derived chiefly from plant and animal extracts, perform the same tasks as their synthetic counterparts, but they are mostly benign, eco-friendly and less harmful to the environment (Kyei *et al.*, 2022; Ong *et al.*, 2021). Pérez *et al.* (2007) have confirmed some protective properties of natural tannin-based bioactive pigments. In their review, Gopikrishnan *et al.* (2015) concluded that natural additives are favorable precursors for developing benign coatings. Idora *et al.* (2015) extracted tannins from *Rhizophora apiculata* and used the extracts as a native fouling resistant medium in epoxy coatings for saltwater applications. They concluded that a low concentration of mangrove polyphenols could exhibit good protective properties. Work done by Byrne *et al.* (2019) established that coatings formulated with tara and quebracho tannins perform better adhesive functions than commercial zinc tetraoxochromate primers.

Manufacturers of paints must consider products that will not compromise the environment, otherwise known as eco-friendly coatings, and yet perform a protective function (Kyei *et al.*, 2020a, 2020b). The question that remains unanswered is whether agro-based coatings could play the same role as other conventional coatings. Some studies have been carried out in the past decade on the valorization of agro-wastes such as cashew nutshells into valuable products (Lochab *et al.*, 2014). Once the coating is applied to a substrate, the corresponding protective function depends on effective drying, adhesion and cohesive strength. An applied coating with insufficient drying, adhesive and cohesive strength is likely to flake off from a substrate – a development that badly impacts the performance of the painted material (Wei *et al.*, 2020). The drying of the applied coating could be physical or chemical depending on the environmental conditions prevailing (Faccia *et al.*, 2009; Xu *et al.*, 2012). Adhesion-enhancing mechanisms between coatings and base substrates must be used when formulating

surface coatings. Some studies (Awaja *et al.*, 2009; Jothi *et al.*, 2020; Wang *et al.*, 2020) have explored mechanisms for improving adhesion. A typical example is the chemical modification of agro-wastes via suitable reactions.

Notably, studies using agro-wastes [viz. peanut skin extract (PSE) and cashew nutshell liquid (CNSL)] in formulating surface coatings are scanty in the literature. The concern has been to find and use safe and environmentally friendly components and precursors in the formulation, which to the best of the authors' knowledge, has not been addressed extensively in the previous studies. Therefore, the current study considers sourcing flavonoids and phenol derivatives from agro-wastes for the synthesis of pigments and binder (resin) as the main precursors in the formulation of surface coatings.

The objective of this study is to use chemically modified PSE as a pigment, CNSL resin as a binder and other natural additives in the formulation of novel surface coatings. The study provides details of the synthesis of the precursors, their characterization and the drying and adhesive properties of the formulated coatings as surface coatings.

2. Experimental

2.1 Materials

All the chemicals were of analytical grade and were acquired from Loba Chemie (Mumbai, India), and were used as they were. Normal hexane (95%), acetone (99.8%), aniline (99.5%), 2-aminophenol (98%) and 4-aminophenol (95%), 2-amino benzoic acid (98%), 4-amino benzoic acid (99.9%), hydrochloric acid (37%), sodium nitrite batch 005914 (99%), sodium hydroxide pellets batch 638903 (98%), methanol (98%), formaldehyde, FeCl₂·4H₂O (99%) and MnCl₂·4H₂O (98%) were used in the extraction of PSEs and synthesis of pigments. Peanut skins and cashew nut shells were obtained from the Ejura market (GPS coordinates: 7.3825549, −1.3770462) in Ghana. Bentonite clay, soyabean oil and toluene were used as additives and solvents, respectively, in the formulation of surface coatings.

2.2 Extraction of phenolics from peanut skin

The roasted skin of peanuts (Runner variety) was pulverized with a multipurpose blender (Vitamix E310 Explorian), filtered through a mesh (Schneider GT107) with 1-mm openings, and defatted with hexane. About 170 g of the skins were soaked in 600 mL of hexane and left to stand for 48 h, after which the hexane was decanted. The defatting was repeated. The defatted peanut skin was oven-dried at 50°C. The extraction of phenolics from peanut skins was done as per a procedure described elsewhere (Kyei *et al.*, 2020a, 2020b) using accelerated solvent extraction conditions in aqueous acetone (50, v/v) as the extraction solvent. The extracts were concentrated using a vacuum rotary evaporator and lyophilized (Cole-Parmer, Vernon Hills, USA). The products were stored at 18°C, pending further analysis. The percentage yield and melting points of the extracts were determined.

2.3 Chemical modification of peanut skin extract (as pigment)

Two diazonium salts were prepared with amino compounds, namely, 2-amino benzoic acid and 4-aminophenol, according to methods described elsewhere (Kovalchukova *et al.*, 2017; Kyei *et al.*, 2020a, 2020b) with slight modifications. Exactly,

1.37 g (0.01 mol) of 2-aminobenzoic acid was dissolved in 45 mL of distilled water containing 12 mL of concentrated hydrochloric acid to make the first diazonium salt. The resulting solution was stirred continuously on an ice bath at 0°C–5°C. A cold, 0.01-M solution of sodium nitrate (0.70 g NaNO₂ in 5 mL of distilled water) was added dropwise to produce a diazonium salt.

An alkaline PSE solution was prepared by dissolving 2.9 g of the extract in a 3% NaOH solution at 0°C–5°C. The alkaline PSE solution was used as a coupling agent for the synthesis of the pigment. The freshly prepared cold diazonium solutions were reacted with the alkaline PSE solution by dropwise addition with effective stirring for 30 min at 0°C–5°C to yield PSE ligands. The solutions were then subjected to vacuum filtration and left overnight to dry in a desiccator. The product, PSE ligand (P1), was bagged and labeled accordingly. The PSE ligand was dissolved in 40 mL of methanol slowly with effective stirring for 15 min. Manganese chloride and iron (II) chloride solutions were prepared by dissolving 0.01 mol of each respective reagent in 20 mL of distilled water. For complexation, the desired metal solution was added slowly to the methanolic solution of the PSE ligand. The resulting solution was then refluxed at 65°C in a round-bottomed flask for 30 min. The complexes were removed via vacuum filtration, and rinsed with water and methanol (1:1) solvent. Finally, the products containing the P2 and P3 complexes (pigments) attained were bagged and labeled accordingly.

The above procedure was repeated in synthesizing a second ligand, P4 with 4-aminophenol as diazonium salt, and two complexes, which were labeled P5 and P6, as shown in Table 1.

2.3.1 Characterization of pigments

FT-IR and UV-visible spectroscopy were used to characterize the pigments. At a wavelength range of 400–4,000 cm⁻¹, infrared spectra were acquired on a Fourier transform infrared spectrophotometer (Bruker Alpha Platinum, ATR, Billerica, USA). The UV-visible spectra of the pigments were recorded in the range of 250–600 nm in methanol solution on a UV-visible spectrophotometer (SPECORD 200 plus–223E1451, Jena, Germany).

2.3.2 Antimicrobial characteristics of pigments

A local company provided the bacteria *Staphylococcus aureus* (*S. aureus*) and *Escherichia coli* (*E. coli*). Stock cultures of bacteria were delivered to the laboratory under the recommended storage conditions. Lysogeny broth (LB) medium with peptone (10 g/L), NaCl (10 g/L) and yeast extract (5 g/L) was used to grow *E. coli*. Regarding *S. aureus* culture, trypticase (tryptic) soy broth medium was made up of pancreatic digest of casein (17 g/L), enzymatic digest of soybean meal (3 g/L), NaCl

(5 g/L), dextrose H₂O (2.5 g/L) and KH₂PO₄ (2.5 g/L). Before using both media, the pH was adjusted to 7.2.

The antibacterial activity of pigments was determined by disk diffusion analysis using *E. coli* and *S. aureus* as typical pathogenic bacteria. LB agar (10 mL) was dispensed into sterilized Petri dishes and solidified within 10 min. The bacterial suspension (*E. coli*) was uniformly inoculated on the solidified agar gel. The pigments were dissolved in methanol. Dropwise, 0.5 mL of pigments (concentration 450 µL) were added to the LB agar plate and incubated at 36°C for 48 h. The procedure was repeated, using *S. aureus* as the bacterium suspension. The antibacterial properties of the samples were compared by the diameter of the zone of inhibition around each disk in duplicate. Reagent blanks were run as a negative control.

2.4 Synthesis of cashew nutshell liquid-modified resin

Cashew nuts were purchased from the Kintampo market (GPS 8°02'60.00N–1°42'59.99"W) located in the Bono East Region of Ghana. Pretreatment and subsequent extraction of cashew nut liquid from the nuts were done as per an existing protocol (Kyei et al., 2019). To synthesize the CNSL-modified binder, a beaker containing 10 mL of CNSL, 15 mL of formaldehyde and 5 mL of 1 M NaOH was homogenized using a magnetic stirrer for 10 min. This was followed by heating under varying temperatures and heating times until a high viscous good surface resin (brown) was obtained (Huo et al., 2018).

2.4.1 Characterization of cashew nutshell liquid-modified resin

The physicochemical properties of the resins obtained were evaluated. The color, hardness, viscosity and solubility were determined as per the protocol used in the earlier work done by Kyei et al. (2019). Comparing these properties to a commercial resin revealed that the CNSL-modified resin meets the required properties.

2.5 Formulation of surface coatings

Paints were formulated on a laboratory scale in a ball mill based on the local raw materials and a modified protocol of Yong et al. (2015). The ingredients, namely, pigment (azo compounds and metal complexes), binder (CNSL-modified resin) and additives (bentonite clay, soyabean oil) were mixed using a high-speed disperser. The materials were allowed to milling into an extremely fine form for a period of 1 h using a ball mill at a speed of 65 rpm. The solvent (toluene) was then added, and dispersion continued for 35 min to allow effective blending. Subsequently, a brown-colored coating was obtained. The procedure was repeated with varying pigment compositions.

Table 1 Designation, components and percentage yields of azo compounds (pigments)

Pigment designation	Coupling component	Diazonium salt used	Metal chloride used	Color	Yield (%)
P1	Peanut skin extract (PSE)	2-Amino benzoic acid	None	Brown	94.66
P2	PSE	2-Amino benzoic acid	FeCl ₂	Deep orange	68.04
P3	PSE	2-Amino benzoic acid	MnCl ₂	Deep orange	47.01
P4	PSE	4-Aminophenol	None	Dark brown	92.71
P5	PSE	4-Aminophenol	FeCl ₂	Deep brown	42.45
P6	PSE	4-Aminophenol	MnCl ₂	Deep red	99.24

2.6 Characterization of coatings

2.6.1 FTIR analyses of coatings

A Bruker Alpha Platinum, ATR, Billerica, USA, with a resolution of 4 cm^{-1} in the absorbance mode, was used to characterize the paints. The samples were retained in an attenuated total reflection accessory with a thermal device and a diamond crystal, and the spectra were recorded in the range of $4,000\text{--}500\text{ cm}^{-1}$.

2.6.2 Determination of drying times

The drying times of the coatings were determined on a concrete panel following the ASTM 1640–95 test method (ASTM D 1640–95, 2008).

2.6.2.1 Dry-to-recoat time. The surface of the concrete panel was cleaned, and a coat of paint was applied. After drying, a second coat of the film was applied to the same position (specified topcoat). The drying times of the first and second applications were recorded as the dry-to-recoat time (ASTM D 1640–95, 2008).

2.6.2.2 Dry-to-touch time. To monitor the drying process, the finger was lightly smeared across the concrete panel. When the coating did not adhere to the finger and did not rub up considerably, it is dried enough to be touched. This was then recorded as the dry-to-touch time (ASTM D 1640–95, 2008).

2.6.2.3 Dry-hard time. Again, the thumb was placed on the concrete panel after applying the paint. A soft cloth was used to polish the contact area after removing the thumb. The concrete panel was checked periodically until the film was dried to a stage at which the pressure that can be exerted between thumb and finger failed to leave a mark behind. The time elapsed was then recorded as the dry-hard time (ASTM D 1640–95, 2008).

2.6.2.4 Dry-through time. Finally, the concrete panel was placed in an airy area after applying the paint. The thumb was then placed on the panel to access the time when it was dried to handle, i.e. the time when the paint was devoid of any proof of deformity. The corresponding time taken was recorded as the dry-through time (ASTM D 1640–95, 2008).

2.6.3 Determination of adhesion

The adhesion test was done according to the ASTM D 3359–97 standard. The paint was first applied to the surface of a concrete panel. A perfect area was selected with the help of a sharp knife with a snipping edge angle of $15^\circ\text{--}30^\circ$ and a spacing dimension of 0.5 mm for measuring individual square cuts. Parallel cuts were made about $3/4$ in and 20 mm long using the cutting edge. Afterward, the film was brushed to get rid of any foreign material. The incisions for the reflection of light from the substrate were inspected to ensure good cutting. The grid area was tapped, providing good contact with the substrate.

After 60 s of application, the tape was detached and examined for any paint removal from the concrete panel. The adhesion was rated per the literature adhesion scale, and their correspondents were recorded (ASTM D 3359–97, 2010).

3. Results and discussion

3.1 Physical characteristics of pigments

The synthesized azo compounds and metal complexes were evaluated as pigments for the formulation of surface coatings. The designation, components and percentage yield of the azo compounds are listed in Table 1. The colors are physical indications of the formation of the azo compounds. The pigments exhibited a very good yield (42.5%–99.24%), which is consistent with yields attained in analogous work by Hawaiz and Samad (2012) and Aravindhnan *et al.* (2014). The melting points (ranging from 265 to >300) are generally high (Table 2) and suggest that the pigments are stable and could withstand heat when deployed in the formulation of coatings. Furthermore, the pigments had wide melting ranges (greater than 5°C). This could be because PSE contains phenolic compounds (flavonoids), which are all capable of coupling reactions with a diazonium salt. As a result, each coupling reaction performed in this study produces a mixture of azo compounds with a wide melting point. All the pigments were soluble in organic solvents (methanol, ethanol and acetone) but insoluble in water (Table 2).

3.2 Spectral analysis of pigments

The IR spectral data of the synthesized pigments are presented in Table 3.

The peaks at $3,186$, $1,600$, $1,491$ and $2,875\text{ cm}^{-1}$ confirm the presence of --OH stretching, aryl substituted $\text{C}=\text{C}$, $\nu(\text{N}=\text{N})$ and --C--H stretching, respectively, in the pigment P1 (Table 3). Its corresponding metal complex, P2, exhibited bands at $3,348$, $1,602$, $1,491$ and $2,900\text{ cm}^{-1}$ because of $\nu(\text{OH})$ group, aryl substituted $\text{C}=\text{C}$, azo $\nu(\text{N}=\text{N})$ moiety and C--H stretching, respectively, while P3 showed the bands $3,230$, $1,601$, $1,490$ and $2,895\text{ cm}^{-1}$, which are attributable to the stretching mode of --OH group, $\text{C}=\text{C}$ of aromatic groups, azo $\nu(\text{N}=\text{N})$ group and --C--H stretching vibrations, respectively. The stretching of metal-oxygen and metal-nitrogen for iron and manganese complexes exhibited bands at 470 and 419 cm^{-1} and 491 and 446 cm^{-1} , respectively. It must be noted that the complexation was through the nitrogen of the azo moiety and hence the lower wavelength (Yousif *et al.*, 2017).

The FTIR spectra of pigment P4, as shown in Table 3, are as follows (KBr cm^{-1}): $3,270[\nu(\text{OH})]$, $1,593[\nu(\text{C}=\text{C})_{\text{Ar}}]$, $1,510$

Table 2 Melting points and solubilities of azo compounds (pigments)

Pigment designation	Melting points ($^\circ\text{C}$)	Solubility			
		Water	Methanol	Ethanol	Acetone
P1	285–298	Insoluble	Soluble	Soluble	Soluble
P2	>300	Insoluble	Soluble	Soluble	Soluble
P3	>300	Insoluble	Soluble	Soluble	Soluble
P4	265–289	Insoluble	Soluble	Soluble	Soluble
P5	277–295	Insoluble	Soluble	Soluble	Soluble
P6	>300	Insoluble	Soluble	Soluble	Soluble

Table 3 Spectral data for pigments

Pigment designation	IR (KBr) (cm^{-1})
P1	3,186b (OH), 1,600 (C=C) aryl, 1,491 (N=N), 2,875 (C–H)
P2	3,348b (OH), 1,602 (C=C) aryl, 1,491 (N=N), 2,900 (C–H), 470 (M–O), 419 (M–N)
P3	3,230b (OH), 1,601 (C=C) aryl, 1,490 (N=N), 2,895 (C–H), 491 (M–O), 446 (M–N)
P4	3,270b (OH), 1,593 (C=C) aryl, 1,510 (N=N), 2,923 (C–H)
P5	3,347b (OH), 1,598 (C=C) aryl, 1,510 (N=N), 2,924 (C–H), 465 (M–O), 442 (M–N)
P6	3,243b (OH), 1,598 (C=C) aryl, 1,509 (N=N), 2,922 (C–H), 467 (M–O), 418 (M–N)

Note: b = broad band

[$\nu(\text{N}=\text{N})$] and 2,923[$\nu(\text{C}-\text{H})_{\text{Aliph}}$]. The first corresponding complex, P5, showed the following spectra (KBr cm^{-1}): 3,347 [$\nu(\text{OH})$], 1,598[$\nu(\text{C}=\text{C})_{\text{Ar}}$], 1,510[$\nu(\text{N}=\text{N})$], 2,924[$\nu(\text{C}-\text{H})_{\text{Aliph}}$], 465 [$\nu(\text{M}-\text{O})$] and 442[$\nu(\text{M}-\text{N})$]. P6 has the following spectra (KBr cm^{-1}): 3,243[$\nu(\text{OH})$], 1,598[$\nu(\text{C}=\text{C})_{\text{Ar}}$], 1,509[$\nu(\text{N}=\text{N})$], 2,922[$\nu(\text{C}-\text{H})_{\text{Aliph}}$], 467 [$\nu(\text{M}-\text{O})$] and 418[$\nu(\text{M}-\text{N})$].

3.3 Antimicrobial activity of pigments

The results of the evaluation of the synthesized pigments against *E. coli* and *S. aureus* are presented in Table 4.

Generally, the pigments showed better antimicrobial activity against *E. coli* than *S. aureus*. Based on the above results, the zone of inhibition range shows a sequence as follows: P4 = P5 > P1 = P2 > P6 > P3 for *E. coli* and P1 = P4 > P2 for *S. aureus*, with P3, P5 and P6 showing no inhibition. The intense inhibition of *E. coli* could be attributed to the presence of the nitro group in the recurring aromatic rings in azo compounds (Mohamed et al., 2020). Besides, the pigments are from natural compounds or extracts from plants, which are reported to have antimicrobial activity (Huang et al., 2021). Microbial activity can alter the characteristics of biofilm, causing corrosion to speed up or slow down in a specified environment. *E. coli*, a sulfate-reducing bacteria, causes microbiologically influenced corrosion by consuming the coating, thereby changing the pH and color of the coating. An enhanced antimicrobial activity observed in P6, P1, P2, P4 and P5 against *E. coli* confirms the pigments' potential as antimicrobial agents and as inhibitors to microbiologically influenced corrosion in surface coatings. Thus, the coatings, when applied could be highly susceptible to *E. coli* degradation and could be a way to prevent microbial proliferation, or at least significantly reduce the number of microorganisms that grow on the substrates (Verdier et al., 2014). We, therefore, suggest that improving the formulation, i.e. varying the proportions, could increase the efficacy of the coatings.

3.4 Physico-chemical characteristics of the resin

The FTIR spectrum of the main precursor for the resin, CNSL, is presented in Figure 1. The presence of a typical “polymeric” –OH stretch is attributable to a peak at 3,407 cm^{-1} and is preceded by a 3,078 cm^{-1} Ar C–H stretch. The presence of the C = C hydrocarbon moiety, C–H asymmetric and symmetric vibrations are because of 3,008, 2,924 and 2,853 cm^{-1} , respectively. The following peaks are worth noting: C = C Ar peaks are represented by 1,604 and 1,450 cm^{-1} and 993 and 910 cm^{-1} , which are typical of *cis-trans* C = C and terminal vinyl groups, respectively.

Table 5 also shows the composition reaction conditions and other properties of the synthesized resin.

In the presence of sodium hydroxide (alkali), a dark brown, highly viscous CNSL–formaldehyde resin was synthesized at an optimal temperature and reaction time of 75°C and 60 min, respectively. The formation could be attributable to the finest crosslinking in the polymer backbones of resins. Curing was

Figure 1 FTIR of cashew nutshell liquid

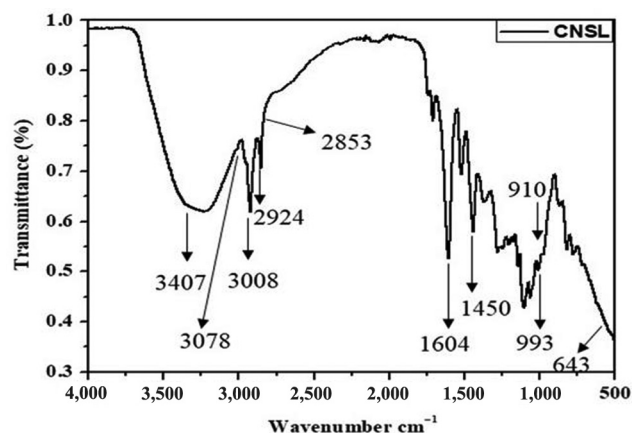


Table 4 Antimicrobial effects of pigments

Pigment designation	Zone of inhibition (mm)	
	<i>Escherichia coli</i>	<i>Staphylococcus aureus</i>
P1	6.0 ± 0.64	8.0 ± 0.23
P2	6.0 ± 0.42	3.0 ± 0.77
P3	4.0 ± 1.02	0
P4	10.0 ± 0.38	8.0 ± 1.16
P5	10.0 ± 0.78	0
P6	5.0 ± 0.89	0

Table 5 Composition and other properties of cashew nutshell liquid resin

CNSL:CH ₂ O: NaOH (mL)	Reaction time (min), temp (°C)	Nature of resin	Color	Flexibility	Solubility	
					Acetone	Toluene
10:15:5	45, 95	Firm	Dark brown	Poor	Soluble	Soluble
10:15:5	60, 75	Highly viscous, flexible	Dark brown	Excellent	Soluble	Soluble
10:15:5	75, 120	Solid	Darker	Poor	Soluble	Soluble
10:15:5	90, 85	Tough, semi-viscous	Dark brown	Good	Soluble	Soluble

dependent on time and temperature. A longer curing time and higher temperature resulted in a highly solidified (hard) darker resin with poor flexibility. This suggests that, generally, the hardness of resins depends on the extent of crosslinking. In all cases, the resins were soluble in acetone and toluene.

3.5 Formulation of coatings

Six surface coatings have been formulated using local and natural raw materials (agro-wastes) as pigments and binders (resin). Based on the pigments P1, P2, P3, P4, P5 and P6, the coatings and compositions were coded as CP1, CP2, CP3, CP4, CP5 and CP6, respectively (Table 6). It has been revealed that such coatings are better substitutes for commercial, non-degradable coatings (Acevedo *et al.*, 2013). This is because coatings formulated from natural precursors are eco-friendly, readily available and cost-effective. The application of agro-wastes as pigments and resins in coatings formulation meets the global anti-pollution requirements (Tadros, 2005), a concept known as waste valorization.

3.6 Spectral analysis of formulated coatings

The structural features associated with the formulated coatings as corroborated by FTIR analysis are presented in Figures 2 and 3. In Figure 2, the FTIR spectra of CP1, CP2 and CP3 indicated the main functional group bands and clearly showed the differences in peak intensity for the functional groups. Based on the bands found on CP1, the following functional groups were assigned: C–H sp³ stretching vibration at 2,917 and 2,850 cm^{−1}, conjugated C=O vibration at 1,741 cm^{−1}, C–H sp³ bending vibration at 1,576 and 1,540 cm^{−1}, C–O vibration at 1377 cm^{−1}. These agreed with previous studies (Syuhada *et al.*, 2020). In spectrum CP2, characteristic peaks around 2,920 cm^{−1}, 2,851 cm^{−1} (CH– in methylene group), 1,710 cm^{−1} (C=O in amide group) and 1,579 cm^{−1} (C = C in benzene ring) were observed (Figure 2). The most important peaks identified in CP3 were distinctive peaks around 3,008 cm^{−1}, 2,923 cm^{−1}, 2,853.04 cm^{−1} (CH in methylene group), 1,602, 1,500 cm^{−1} (C = C in benzene ring). Moreover, other absorbance peaks around 3,357 cm^{−1} (OH– groups) and 1,708 cm^{−1} (CO= in saturated aldehyde, ketone or

acid) showed up. The pigment used in this study contained an azo v(N = N) compound. The small peaks around ~1,464 cm^{−1}, 1,439 cm^{−1} and 1,441 cm^{−1} were attributed to the azo v(N = N) bond in CP1, CP2 and CP3, respectively. The results confirmed the formation of the target coatings.

The IR spectra of the formulated coatings CP4, CP5 and CP6 (presented in Figure 3) are pretty similar to Figure 2. The spectrum CP4 shows the occurrence of important linkages such as the OH– group at a strong transmission band at 3,367 cm^{−1} and the carbonyl group at 1,604 cm^{−1}. There is also a stretching band around 1,382 cm^{−1} because of C–(CH₃)₂ and a stretching band at 1,015 cm^{−1} because of the C–O group. Moreover, a band at 1,267 validates the presence of a methylene group in the structure, while the bands at 2,922 and 2,852 cm^{−1} are characteristics of aliphatic –C–H (Tawfik *et al.*, 2017). Similar trends were observed for CP5 and CP6. In CP5, the two intense peaks at 2,291 and 2,852 cm^{−1} are from the primary C–H stretch peaks of methyl groups. Moreover, a broad peak around 3,361 cm^{−1} could be attributed to OH groups affected by hydrogen bonding. The occurrence of CO vibration at 1,006 and 1,591 cm^{−1} (C = C in the benzene ring) could be observed. CP6 presents similar peaks, affirming the successful formulation of coatings from CNSL and PSE.

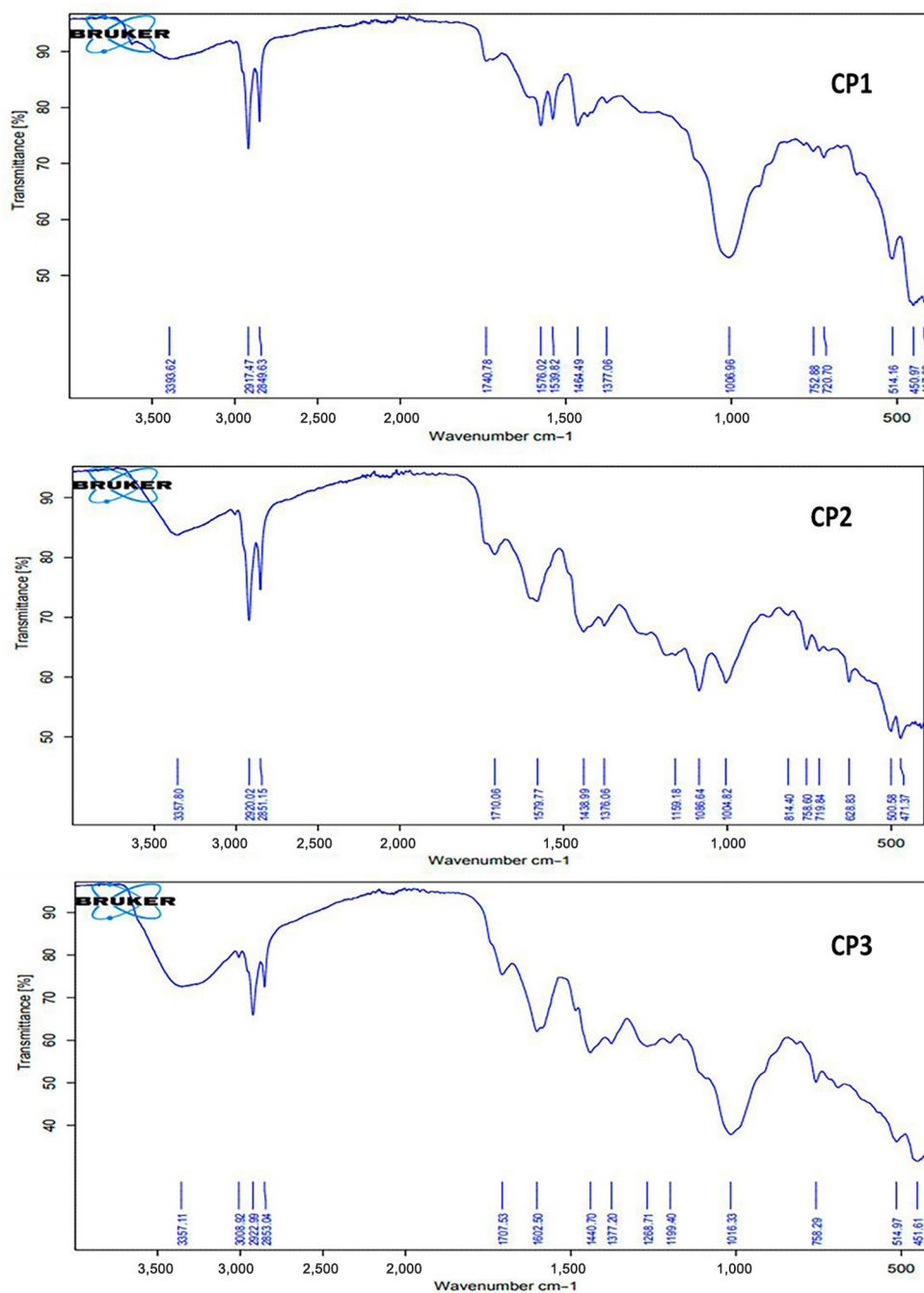
3.7 Composition, color and percentage yields of coatings

The composition, color and percentage yields of the coatings are presented in Table 6. Although the composition of the coatings is not the same, each of them contains the major components of a pigment, a binder (resin) and low levels of other additives dissolved in a vehicle (solvent). The incorporation of local raw materials as pigment and binder (resin) in this study is a sustainable approach to developing green materials with no or less dangerous influence on the environment. The color of the products was the same as the color of the pigments used in the formulation. The coatings gave a yield (ranging 73%–86%), which is acceptable and comparable to those reported in the literature (Escobar *et al.*, 2019).

Table 6 Composition, color and percentage yield of coatings

Coating designation	Composition	Color	Percentage yield (%)
CP1	P1, CNSL resin, Solv, Addit.	Brown	86
CP2	P2, CNSL resin, Solv, Addit.	Deep orange	81
CP3	P3, CNSL resin, Solv, Addit.	Deep orange	73
CP4	P4, CNSL resin, Solv, Addit.	Dark brown	86
CP5	P5, CNSL resin, Solv, Addit.	Deep brown	76
CP6	P6, CNSL resin, Solv, Addit.	Deep red	77

Notes: Solv., solvent; Addit., additives (variable proportions of bentonite clay, soybean oil)

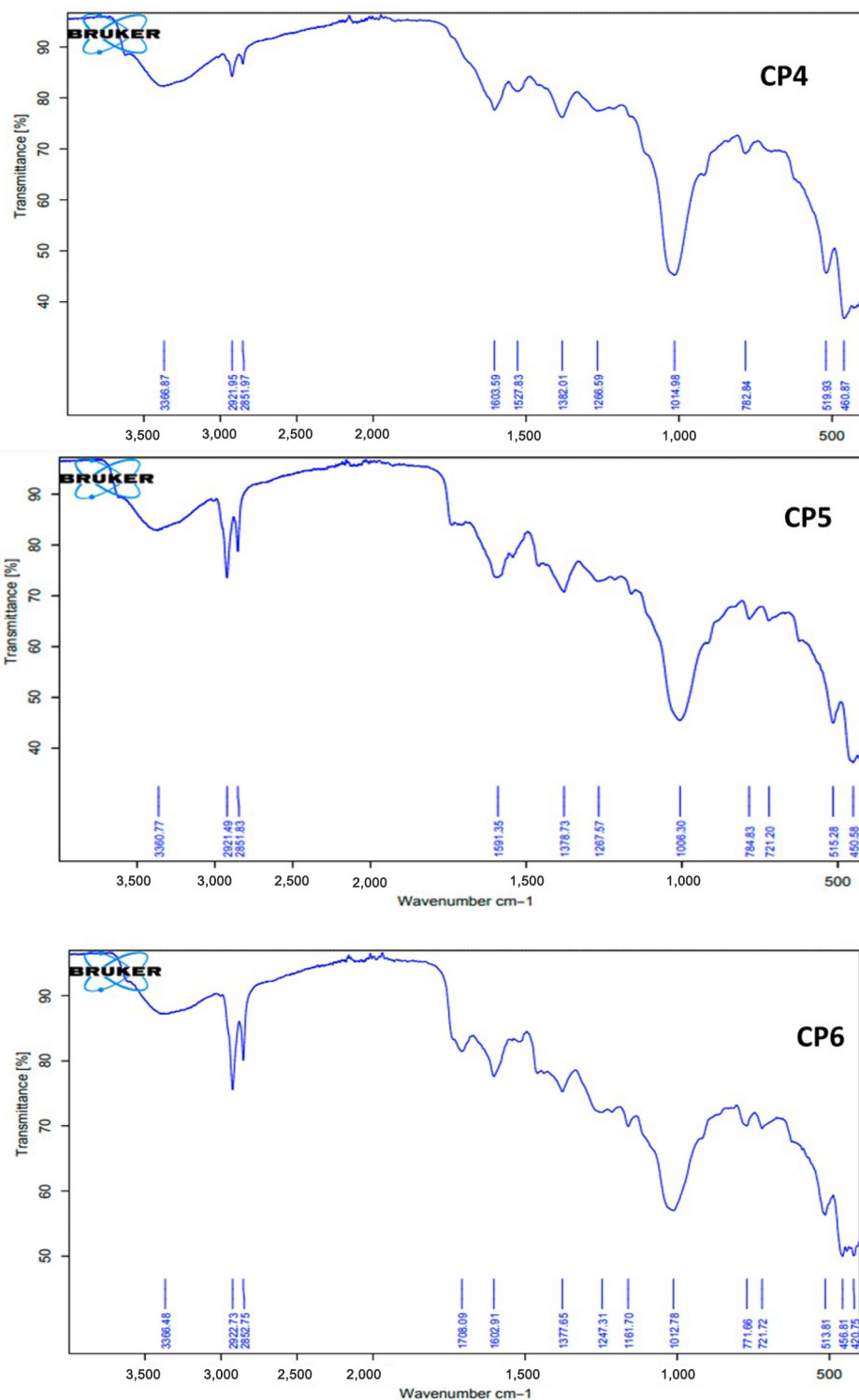
Figure 2 FT-IR spectra of coatings, CP1, CP2 and CP3

3.8 Drying time of coatings

The drying time of coatings such as dry-to-recoat, dry-to-touch, dry-hard and dry-through is presented in Table 7. A paint's performance is dependent on its physicochemical parameters, including its drying time (Cruz *et al.*, 2019). The data reveal relatively short dry-to-recoat times (ranging between 0.33 and 1.50 min) and short dry-to-touch times (ranging from 3.41 to 60.03 min), a short dry-hard time of 4.41–61.33 min and a longer dry-through time of 5.56–80.14 min. Moreover, CP2 and CP4 had higher drying times

(Table 7) than the other samples. The inconsistencies in drying times could be attributed to the varying compositions of the coatings.

The shorter curing times may be attributable to the evaporation of volatile compounds. Thus, the physical drying usually takes place in a short period. The addition of dryer to the components of paints typically acts as catalysts to drastically reduce the drying time via an auto-oxidation effect in the film formation (Križan *et al.*, 2019). In this study, soybean oil was used as a dryer, which might have contributed to the shorter

Figure 3 FT-IR spectra of coatings, CP4, CP5 and CP6

drying times compared to literature (Akaranta and Aloko, 1999; Ikeda *et al.*, 2002). The dryer might have accelerated the drying process, making the chemical oxidative process faster than physical drying. The double bonds in the dryer are oxidized into single bonds, increasing the molecules' reactivity (Cruz *et al.*, 2019; Simoes, 2015).

3.9 Adhesion of coatings

Table 8 presents the results of the cross-cut adhesion test after five days of drying. The coating-coded CP3 had 15%–35% of the grid detached per the ASTM classification. The paint film peeled off at a point on the boundary and part of the squares after removing the tape from the substrate, and the adhesion

Table 7 Drying times of coatings at 29°C

Coating designation	Dry-to-recoat (min)	Dry-to-touch (min)	Dry-hard (min)	Dry-through (min)
CP1	0.47	6.49	9.30	25.22
CP2	1.08	14.06	15.20	46.02
CP3	1.50	5.09	6.33	10.05
CP4	1.11	60.03	61.33	80.14
CP5	0.33	3.41	4.41	5.56
CP6	1.25	5.24	6.01	15.32

Table 8 Adhesion test results of coatings after five days of drying

Coating designation	Adhesion (% area removed)	ASTM classification
CP1	5%–15%	3
CP2	Less than 5%	4
CP3	15%–35%	2
CP4	Less than 5%	4
CP5	Less than 5%	4
CP6	Less than 5%	4

Table 9 Properties of Temac TMP 149 coating

Parameter	Standard
Drying time	
Touch dry	30 min at 20°C
Hard dry	6 h at 20°C
Adhesion	Excellent

was “fair” in this case (ASTM D 3359–97, 2010). This could have been caused by the delamination of the coating from the substrate, which creates a breach between the surface and the coating, allowing water, oxygen, and soluble salts to interact (Sivakumar *et al.*, 2021). The remaining samples exhibited “good” (CP1) and “very good” (CP2, CP4, CP5 and CP6) responses to the substrate. This indicates that the coating had an excellent cohesive bond and adhered well to the substrate. The degree of reactivity of the organofunctional group (from the azo compound) with the modified CNSL resin is directly proportional to the coating’s enhanced adhesion strength, resulting in a strong connection at the coating’s interface (Cicek *et al.*, 2021).

These results (Table 8) are consistent with the findings of Cruz *et al.* (2019) and Chen *et al.* (2017). Adhesion is significant in the protective performance of coatings. Coating adhesion strength to a substrate is important for functional applications. The results obtained indicate that adhesion between the first and the substrate was stronger than the cohesion between the first and the second coating. In the case of a base material to which a coating is applied, “excellent adhesion” could be attributable to a strong hydrogen bond between the coatings’ functional group and the surfaces they are applied to or hydroxides (Ramesh *et al.*, 2013).

According to Mohamed *et al.* (2010), excellent and very good adhesion on substrates is a fundamental prerequisite for high corrosion inhibition performance. Therefore, coatings

CP2, CP4, CP5 and CP6 might be suitable corrosion inhibitors in oilfields. Coatings with high adhesion strengths can give superior corrosion protection. On the other hand, poor adherence may hasten the corrosion process and compromise the coating’s longevity (Ren *et al.*, 2022). Thus, the application of these coatings (which exhibit antimicrobial activity against sulfate-reducing bacteria) could perform protective functions such as prohibiting water, oxygen and soluble salts from penetrating as long as they adhere to the substrate base, thereby resisting corrosion (Cho *et al.*, 2019; Wang *et al.*, 2020). Because long-term corrosion protection is dependent on the coating’s integrity, the prevailing and reasonable assumption is that enhancing adhesion will extend the life of the corrosion protection (Croll, 2020). Subsequently, the substrate’s enhanced durability will arise from a lesser environmental impact.

Table 9 presents the drying time and adhesion properties of a benchmark commercial coating (Temac TMP 149).

A comparison of our results with a commercial coating (Temac TMP 149) in Table 9 shows that the surface coatings possess an analogous drying time and adhesive properties. This means our product compares well with current industrial standards, in addition to the superior properties exhibited by the synthesized pigment and resin. The eco-friendliness of the coatings could be improved upon in future research by using no or low volatile organic compounds in the formulation.

4. Conclusion

Surface coatings have been formulated using natural precursors, namely, PSE-azo-metal complexes as pigments, CNSL resin as a binder, soybean oil as a dryer and bentonite clay as an anti-settling agent. Very promising results have been obtained concerning the characterization of bio-based pigments and resins. The pigments could be used as antibacterial agents and corrosion inhibitors in surface coatings because they have good antimicrobial action against *E. coli*, a Gram-negative bacterium. The CNSL-resin exhibited the required properties for application as a precursor for the synthesis of coatings, friction materials, surfactants and as an intermediate for synthesizing other resins. We, therefore, deduce that the superior attributes of the binder and resin are attributable to the chemical modification method deployed. In effect, this study has offered a novel solution for the formulation of bio-based coatings to conform to the global drift of using natural resources rather than petroleum resources.

The coatings formulated exhibited drying time and adhesion properties comparable to a commercial coating (Temac TMP 149), and in the literature. Our findings affirm that adhesion between coatings and a substrate base plays a crucial role in the protection performance. The formulation is novel, cost-effective and suggests the possibility of developing coatings from natural raw materials that do not cause environmental change. Further characterization of the formulated coatings would include surface morphology, thermal stability and corrosion behavior.

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