

COPPER OXIDE NANOPARTICLES AS AN EFFICIENT CO-CATALYST FOR NEEM SEED OIL-BASED ALKYD RESIN SYNTHESIS

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ABSTRACT

This study explores the usefulness of CuO nanoparticles as a co-catalyst in the synthesis of neem (*Azadirachta indica*) seed oil-based alkyd resin (NSOAR). CuO nanoparticles were synthesised by the sol-gel method. A dual catalytic system of PbO and CuO nanoparticles was used to catalyse the reaction. Acid value was determined over time, which indicated that the rate of reaction was enhanced substantially due to the presence of CuO nanoparticles. The synthesised alkyd resin was characterised by Fourier transform infrared spectroscopy, thermal analysis, and gel permeation chromatography. This study was aimed to explore a modified catalytic route in the production of neem seed oil-based alkyd resin. The dual catalytic system enhanced the reaction kinetics, improved the degree of polymerisation, and improved the physicochemical properties of the alkyd resin. The presented study is closely connected to the SDG-9: Industry, Innovation and Infrastructure.

Keywords: Alkyd Resin, Catalyst, Nanoparticle, Acid Value, Coating

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INTRODUCTION

The global coating industries are facing growing sustainability challenges as the conventional alkyd resin synthesis heavily relies on petroleum-derived raw materials, that have an adverse effect on the environment. This necessitates a strategic transition towards alternative bio-based sustainable feedstocks that offer environmental sustainability and economic feasibility. Plant seed-derived oils have gained significant attraction due to their abundance and economic viability.¹ For alkyd resin synthesis, vegetable oils like soybean (*Glycine max*) oil, sunflower (*Helianthus annuus*) oil, linseed (*Linum usitatissimum*) oil, oleander (*Thevetia peruviana*) oil, and chia (*Salvia Hispanica L.*) oils are used for polymer synthesis applications, primarily attributed to their cost-competitive nature, widespread accessibility, and inherent biodegradation properties.²⁻⁷ Non-edible plant oils, being unaffected by the supply-demand constraints and price volatility associated with edible oils, have emerged as promising alternatives and consequently have attracted considerable research attention in alkyd resin synthesis. Derived through the polycondensation of polyhydric alcohols with dibasic acids and oil-derived components, alkyd resins constitute one of the most important classes of polymeric materials in global coating applications.⁸ Owing to the very good film-forming ability, excellent adhesion, superior durability under versatile environmental conditions, and, most importantly, the economic viability, the alkyd resins are popularly used in coating applications. Generally, a polybasic acid, a polyhydric alcohol, and glycerides are used in the synthesis of these resins.^{9,10} Thus, alkyd resins are prepared using three different methodologies, namely (i) alcoholysis or monoglyceride process, (ii) fatty acid process, and the (iii) acidolysis process.^{11,12} The selection of the method for synthesizing resin mainly depends on the nature of the initial reactants, solvents and their nature.¹³ Amongst these three, the alcoholysis process is the most extensively utilized industrial method owing to its effectiveness, simplicity and steady product quality.¹⁴ Alcoholysis involves two stages, the first step termed the alcoholysis stage, is where triglyceride are transformed to monoglyceride by reacting with polyols. In this stage, the formation of monoglyceride is confirmed by solubilizing the reaction mixture in methanol in a 1:3 ratio and checking that the whole solution is clear.¹³ A clear solution in methanol reveals monoglyceride formation. The next step is called as

polyesterification, where monoglycerides undergo condensation reactions with dibasic acids to form the final product.^{10,15} Monoglyceride formed in the alcoholysis stage maintains homogeneity within the reaction medium and it performs a vital role in the polyesterification stage.^{16,17} The general alkyd resin synthesis routes have undergone continuous improvement over the last few decades in quest of their reliability, quality and economic viability.¹⁸ Nevertheless, the new research studies for developing efficient catalytic systems remain reasonably underexplored.¹⁹ Traditional resin preparation routes mainly employ homogeneous base catalysts like $\text{Ca}(\text{OH})_2$, LiOH , NaOH , KOH etc; however, heterogeneous substitutes, for example CaO , zinc-alumina mixed oxides ($\text{Zn}/\text{Al}_2\text{O}_3$), and CaCO_3 , have revealed potential for moderate temperature resin synthesis ($170\text{--}250^\circ\text{C}$).^{15,20-22} In recent years, the incorporation of nanoscale materials as catalysts have gained attentions because of their ultrafine dimensions, extensive surface areas, and unique catalytic properties.^{19,23} Integrating nanosized catalysts into conventional catalytic systems may boost the polymer properties and improve reaction kinetics.²⁴ *Azadirachta indica* (neem) plants are freely available; seeds possess a good fatty acid composition required for alkyd resin synthesis and are non-edible with comparatively higher oil yield.²⁵ Production of bio-based alkyd resins from such renewable resources contributes to the larger objectives of the SDGs and sustainability by reducing reliance on petroleum-based synthetic feedstocks. However, limited reports are available on an efficient nano-catalytic route for synthesis of alkyd resins based on plant seed oils, which can directly contribute to the practical application of these renewable raw materials. Therefore, this study explores the synthesis and catalytic performance of CuO NPs as co-catalysts in conjunction with PbO for synthesising NSOAR via the monoglyceride process i.e., the alcoholysis synthesis route. This research aims to evaluate the catalytic efficiency of the dual catalytic system, monitor reaction kinetics through acid value determination, and assess the anticorrosion properties of the synthesised NSOAR.

EXPERIMENTAL

Material and Methods

Neem (*Azadirachta indica*) seeds were collected from the vicinity of Azara, Guwahati, Assam, India. The preparation of seed powder involved drying the seeds under sunlight, followed by grinding using a mechanical grinding machine. Chemicals and reagents used in this work were of analytical grade and further purification of these was not considered. Among the chemicals and reagents, AR-grade phthalic anhydride and glycerol were procured from ‘Merk’ (Mumbai, India) and copper acetate was obtained from ‘Loba chemie’ (Mumbai, India). NaOH and n-hexane were obtained from ‘HiMedia’ (Mumbai, India).

Extraction of Neem Seed Oil

One of the most common methods used for extracting oils from plant seeds is the Soxhlet extraction method.^{6,7} Herein, petroleum ether was used as the solvent for oil extraction. After each extraction cycle, the solvent was removed using a rotary evaporator to yield a light yellowish-brown oil. The recovered solvent was reused for subsequent extractions. After that, the extracted seed oil was purified by using ‘column chromatography’ having a mobile phase eluent of ethyl acetate/n-hexane with a ratio of 1:9 (v/v).

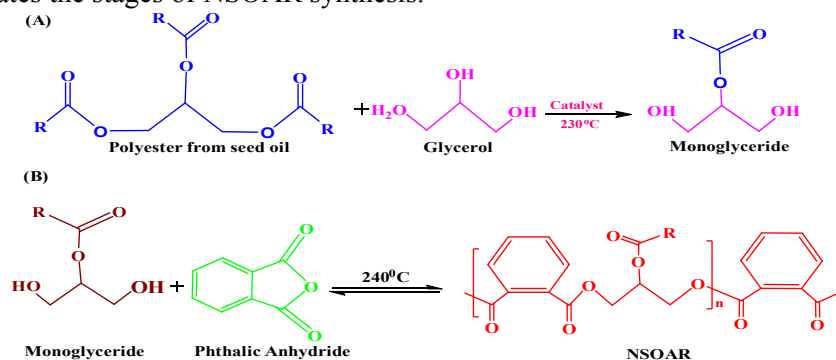


Fig.-1: Neem Plant from Azara, Guwahati, Assam

Synthesis of CuO NPs and NSOAR

For the synthesis of CuO NPs, the sol-gel method was followed. Specifically, 0.1997g (1 mmol) of $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$ was solubilized in 100ml of double-distilled water and stirred to make a 10mM

solution. To this precursor solution, dilute NaOH solution (0.10 M) was added and stirring was carried out for 1 hour at 70°C. After continuous stirring black precipitate appeared. The precipitate was centrifuged at 8000rpm for half an hour and collected. The nanoparticles collected as precipitate were washed with water and dried. Finally, the dried particles were calcinated at 400°C for 1 hour. The resin NSOAR was synthesized by using the most common synthetic route, the alcoholysis-polyesterification method, with PbO acting as a catalyst, and CuO nanoparticles (CuO NPs) were utilized as a co-catalyst (Scheme-1). In the alcoholysis stage, oil weighing 17.43 g (18 mmol) was mixed with 3.68g (40 mmol) of glycerol, and then PbO (5mg) was added as a catalyst under controlled conditions (230°C, 1000 rpm, 1 h).²⁵ The polyesterification method was initiated by lowering the temperature to 120°C for the addition of phthalic anhydride weighing 8.88g (60 mmol), and then further reaction was carried out at 240°C. Scheme-1 illustrates the stages of NSOAR synthesis.



Scheme-1: (A) Alcoholysis and (B) Polyesterification Steps Involved in the Synthesis of NSOAR

Characterisation of CuO NPs and NSOAR

The synthesised samples of alkyd resins and CuO NPs were characterised by FTIR spectroscopy, transmission electron microscopy (TEM), DLS study, GPC analysis, thermal study, contact angle measurements and anticorrosion studies. For all the FTIR spectra, a Cary 630 FTIR Agilent Technology spectrophotometer within the 400–4000 cm^{-1} range was used. TEM observations of nanoparticles were made on a JEOL (JEM-2100) operated at 200 kV. For thermal characterisation, standard thermogravimetric methods were applied to assess decomposition behaviour and thermal stability parameters. A Shimadzu TGA-50 analyser, operating in an inert N_2 atmosphere with controlled heating (10°C/min), was used for the analysis and to obtain TGA, DTG, and DTA profiles for extensive thermal evaluation.

Study of Wettability and Anti-corrosion

Surface wettability in water was studied by using water contact angle (WCA) measurements. Thus, it was assessed via sessile drop contact angle measurements (DMs-401 from Kyowa Interface Science, Japan). The anticorrosion performance of the synthesized resins was evaluated by coating mild steel panels having length 11cm and width 6.5cm and subjecting these to immersion tests using 3.5 wt% aqueous sodium chloride solution.²⁶

RESULTS AND DISCUSSION

Characterization of CuO NPs

Morphology and crystallinity of the synthesized CuO NPs were investigated using TEM and selected area electron diffraction (SAED) study. Figures 2a and 2b presented the representative TEM micrographs, which clearly reveal the formation of quasi-spherical CuO NPs. The particles appeared well-distributed, with some minor agglomeration of metal oxide nanoparticles. The SAED pattern in figure-2c revealed clear concentric diffraction rings, showing the crystalline nature of the prepared CuO NPs. However, the hydrodynamic diameter of the particles in the DLS study showed a slightly larger average size as compared to TEM micrographs. The higher hydrodynamic diameter of the particles in DLS can be attributed to the swelling of the particles as well as some agglomeration of the particles in the medium. Analysis by DLS showed that the synthesized CuO NPs exhibited a relatively narrow size distribution, thus showing an average hydrodynamic diameter of 62.95 nm with a PDI of 0.332 (Fig.-2d).

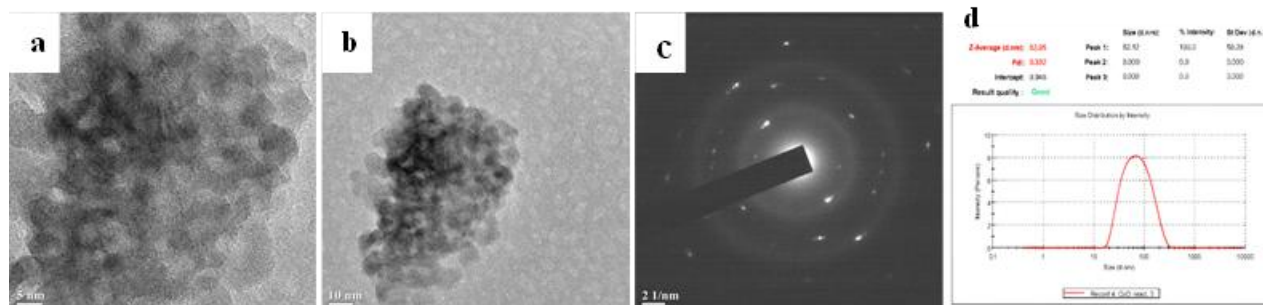


Fig.-2: (a), (b) Representative TEM Micrographs; (c) SAED image; and (d) DLS Profile of CuO NPs

Catalytic Efficiency of CuO NPs

The synthesis of the NSOAR proceeded in two distinct stages: alcoholysis followed by the polyesterification. In the alcoholysis stage, triglycerides are converted into monoglycerides. Triglycerides are non-polar molecules, while monoglycerides are polar molecules. Therefore, their solubility in a polar solvent like methanol is different, and using solubility in methanol, monoglycerides can be distinguished from triglycerides, and the formation of monoglycerides in this stage is confirmed by mixing the reaction mixture with methanol (1:3 ratio) to get a clear solution.¹³ The progress of the reaction and catalytic influence of CuO were observed by determining the acid value of the product at predetermined time intervals (Fig.-3), and the average acid values of triplicate experiments were reported. It was observed that the time required for completion of the monoglyceride formation in the CuO-PbO dual catalyst system was significantly less than that required for the PbO-catalyzed system, indicating efficient catalytic activity of the CuO NPs. As the reaction progresses, a decrease in the acid value of the product with time was observed. As the acid value reached to around 10, the heating was stopped. Scheme-1 illustrates the stages of reactions involved in synthesis of NSOAR.

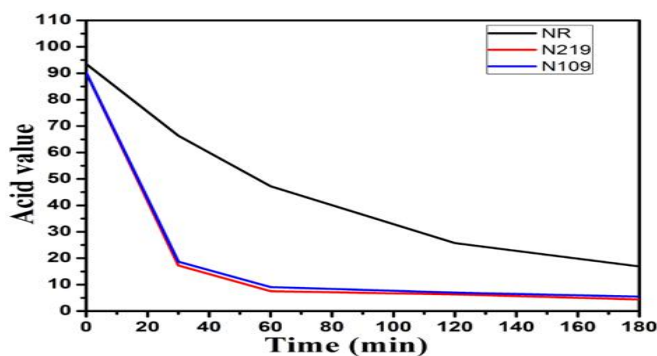


Fig.-3: Acid Value vs Reaction Time Graph

The results demonstrated a consistent decline in acid value as the reaction progresses (Fig.-3); a faster reduction in acid value was observed during the initial stages, whereas in the later stage the decline was lower. This could be explained by the fact that primary and secondary hydroxyl groups react differently with the dibasic acid, leading to different rates of reduction of acid value. In the polyesterification process, the presence of hydroxyl groups can be affirmed by rapid reduction in acid value.²⁷ Whereas the presence of secondary hydroxyl groups is indicated by the relatively slower reduction in acid value.¹⁸

The acid value for the reaction with 0.219 mg of CuO (N219) showed a more pronounced reduction compared to the only PbO-catalyzed reaction (NR). Finally, the N219 system achieved a significantly lower acid value than the NR and N109 systems (sample with 0.109 mg of CuO). This indicates that the CuO NPs facilitate a faster conversion of carboxylic acid groups, driving the esterification equilibrium forward (polyesterification). Moreover, the reaction of N109 showed a slight difference in the acid value as compared to the reaction of N219. To further validate the catalytic efficiency, DP and the extent of reaction, P , were determined using the equation-²⁸

$$P = (C_0 - C_t)/C_0$$

$$DP = 1/(1 - P)$$

Where C_0 and C_t indicate the acid value at initial time (0 minutes) and at time 't' respectively.

The incorporation of CuO NPs resulted in a substantial enhancement in the polymer chain growth. The CuO NPs resulted in substantially higher degrees of polymerisation for N109 and N219 formulations (DP value 16.39 and 20.49, respectively) compared to the control resin NR (DP value 5.52), indicating improved catalytic efficiency. The extent of reaction was also notably enhanced, with P values reaching 0.94 and 0.95 for N109 and N219, respectively, compared to 0.82 for the control resin sample NR. The MW and the polydispersity index (PDI) of the NSOAR prepared (Table-1) for NR and NR219 were tabulated in Table-1. When comparing the values of M_n and M_w for NR and NR219, it further supported the above findings of DP values calculated from acid values.

It was noticed that both number average and weight average molecular weight (M_n and M_w) of the resins NR and NR219 increased as the reaction progressed. Various reports have been reported for seed oil alkyds, which supported the observation cited above.^{29,30} In general, for both the samples, the increase in extent of the reaction, P, increased M_n and M_w . Similarly, PDI was also found to increase slightly in the latter part of the reaction. Moreover, if we specifically compare the trend of increase in molecular weights (M_n and M_w) of NR and NR219, it was clear from Table 1 that the increase in molecular weight (M_w or M_n) was higher in the N219 samples (with different time intervals) compared to NR samples. These results confirmed that CuO NPs function effectively as co-catalysts, accelerating the esterification reactions and promoting faster achievement of higher molecular weight alkyds.

Table-1: DP and P of the Synthesised NSOAR

Alkyds	C_0	C_t	P	DP
NR	93.5	16.93	0.8189	5.5218
N219	90.2	4.4	0.9512	20.4918
N109	90.2	5.5	0.939	16.39

C_0 = acid value at time 0 min; C_t = acid value at time 180 min

Table-2: M_w , M_n and PDI of NSOAR Designated as NR and N219 at different reaction times

Time (min)	NR			NR219		
	M_w (g/mol)	M_n (g/mol)	PDI	M_w (g/mol)	M_n (g/mol)	PDI
0	2077	2005	1.036	2116	2042	1.036
30	2097	2016	1.040	2130	2056	1.036
60	2109	2035	1.036	2437	2275	1.071
120	2335	2178	1.072	2793	2470	1.131
180	2384	2176	1.096	2803	2430	1.153

FTIR Analysis

FTIR analysis revealed characteristic absorption bands that confirmed the formation of the resin (Fig.-4). When the neem seed oil (NO) was examined, absorption bands responsible for the aliphatic C–H stretching at $2857\text{--}2925\text{ cm}^{-1}$; a sharp band at 1749 cm^{-1} assigned for carbonyl stretching of triglyceride; a band for C–H bending of the methylene group was observed at 1467 cm^{-1} ; and the absorption band at 1166 cm^{-1} responsible for the ester C–O stretching were observed. An additional peak at 719 cm^{-1} was observed and might be due to unsaturated fatty acid.^{25,31}

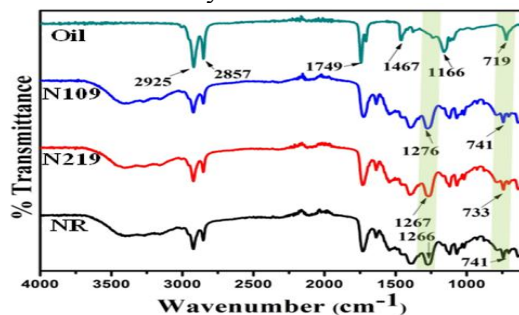


Fig.-4: FTIR Spectra of Oil and NSOARs

FT-IR spectra for the seed oil NO, NR, N109, and N219 were also presented in Fig.-4. The appearance of a new absorption band, due to the ester linkage formed in the polyesterification step of the alkyd synthesis, at $1276\text{--}1266\text{ cm}^{-1}$, which was absent in the spectrum for NO, provides evidence for the formation of NSOAR.

Thermal Analysis

The thermogravimetric analysis of the synthesised NSOAR demonstrated good thermal stability. This provides insights into thermal behaviour and potential applications of the resin in coatings. The TGA, DTG and DTA thermograms of NSOARs were presented in Fig.-5, Fig.-6 and Fig.-7 respectively. Slight enhancement of thermal stability was observed in NR109 and NR219 samples compared to the NR sample (Fig.-6). DTG and DTA profiles revealed that both NR and N109 showed a broad single-step decomposition, while the N219 resin exhibited two-step decomposition with maxima at 381°C and 408°C , which indicated that the CuO NPs are delaying the breakdown of the polyester backbone.³²

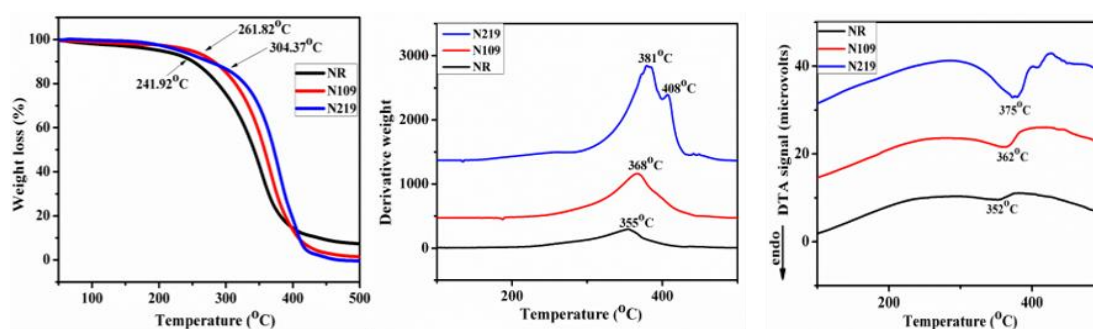


Fig.-5: TG Curve of NSOARs. Fig.-6: DTG Thermogram of NSOARs. Fig.-7: DTA Curve of NSOARs

Water Contact Angle Measurement

Surface wettability of the NSOARs was analysed through water contact angle (WCA) measurements to assess the hydrophobic properties and potential coating performance of the synthesized resins (Fig.-8). We measured contact angles of different resins NR, N109 and N219 to understand how nanoparticle incorporation affects the surface behaviour, and it was found that water contact angles ranged from 72.2° to 102.8° . Sample N109 (WCA value 94.50) showed enhanced hydrophobic character compared to the control resin NR (72.2°), while the sample N219 demonstrated better hydrophobicity and the highest contact angle value (102.8°).

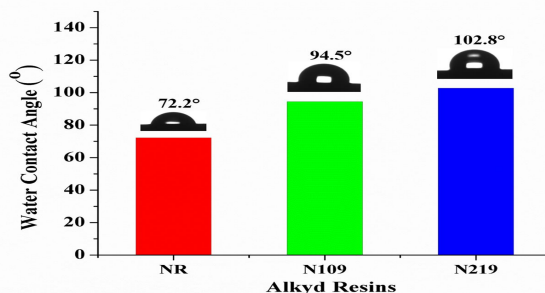


Fig.-8: Water Contact Angle of NSOARs

Anticorrosion Performance

Figure-9 illustrates the visual changes in the coated panels over a period of 6 days. After 4 and 6 days of immersion, the plates were inspected. The uncoated panel showed severe corrosion and rust accumulation. Also, the panel coated with NR displayed significant blistering and rust formation. In contrast, the extent of rusting on the N219-coated panel was negligible. It was observed that the steel plate coated with resin N219 exhibited superior anticorrosive properties compared to all other panels. The increasing order of corrosion resistance was $\text{N219} > \text{N109} > \text{NR}$.

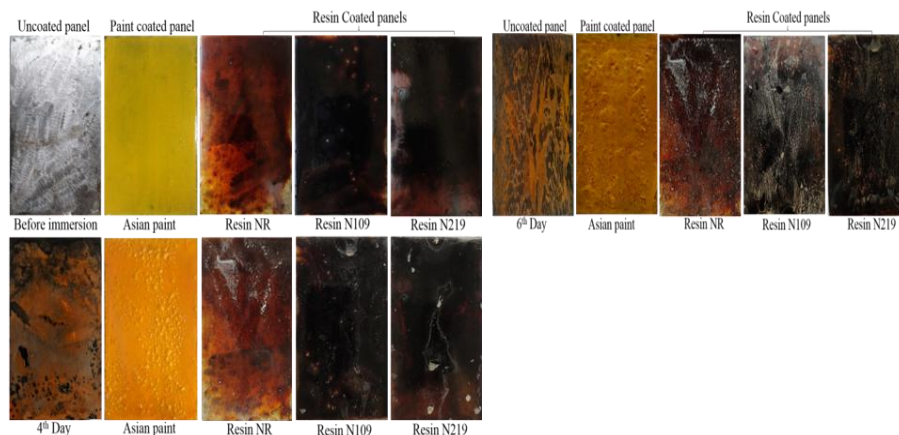


Fig.-9: Study on Corrosion Resistance of different NSOARs

The enhanced corrosion resistance in N219 could be attributed to two factors. First, the presence of CuO NPs enhanced the hydrophobicity of the coating (as evidenced by the increased water contact angle of 102.8°), which effectively repelled water and aggressive ions from the metal surface. Second, the catalytic effect of CuO resulted in a higher degree of polymerisation. This formed a compact network and acted as a robust physical barrier, making the diffusion path for corrosive agents more tortuous and difficult, thereby significantly delaying the onset of corrosion. Overall, the use of the renewable feedstock to synthesize alkyd resin and the application of CuO NPs provide a new catalytic route for preparing NSOARs.

CONCLUSION

This study successfully demonstrated the efficacy of synthesized CuO NPs as a high-performance co-catalyst for the synthesis of sustainable NSOARs. The integration of CuO into the PbO-catalyzed system significantly increased the degree of polymerisation and molecular weight. Beyond catalysis, the in-situ incorporation of CuO yielded a nanocomposite with better physicochemical properties compared to the resin without it. The cured films exhibited enhanced hydrophobicity, with water contact angles rising from 72.2° to 102.8° . The NSOARs have also exhibited notable resistance to corrosion. Thus, CuO co-catalysed neem seed oil-based alkyd resins possessed great potential as a robust and eco-friendly candidate for industrial protective coating applications, contributing to global sustainability and SDGs.

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CONFLICT OF INTERESTS

All authors state no competing financial or other interests.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-9: Industry, Innovation and Infrastructure*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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