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Green synthesis of Metal Oxide nanoparticles and their catalytic activity

for the reduction of aldehydes

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Graphical Abstract



Highlights

- Metal Oxide (MO) nanoparticles were synthesized using a green synthetic method and characterized.
- > The catalytic property of the synthesized MO nanoparticles were examined.
- > A green protocol for the reduction of aromatic aldehydes is demonstrated.

ABSTRACT

In the present work, a green synthesis of Metal Oxide nanoparticles was demonstrated using the freshly prepared aqueous extract of the immature fruit of *Cocos nucifera* and the MO nanoparticles were characterized by the analytical techniques such as UV-Vis, FT-IR, XRD, SEM, TEM and EDAX. Characterization techniques confirmed that the biomolecules involved in the formation of nanoparticles and also they stabilized the nanoparticles. The synthesized MO nanoparticles were used as catalysts for the reduction of aromatic aldehydes. The reduction was done at mild reaction conditions using ammonium formate as a green hydrogen donor and

the corresponding alcohols were obtained in 2-24 h with excellent yields. The reduction reaction was optimized using various solvents, loading of catalyst and at different temperatures.

Keywords: Cocos nucifera, metal oxide nanoparticles, reduction, aldehyde, nanocatalysis.

1 Introduction

The field of nanoscience and nanotechnology is growing and glowing in a high way because of its applications in various fields [1]. In the ancient methods, the nanoparticles are synthesized and stabilized by the stabilizing agents to prevent aggregation [2-4]. Nowadays, green synthetic route is used to synthesize nanoparticles in which the biomolecules can act both as reducing and stabilizing agents [5-7]. Commonly, biomolecules like amino acids, plant extracts, microorganisms and enzymes are used to synthesize nanoparticles in green chemistry [8-9]. Synthesis of nanoparticles using plant materials is gaining more attraction from the researchers, due to its simple experimental procedures. Various plant materials are used for the synthesis of nanoparticles which may be in the form of zero valent [10,11], metal [12-15] or metal oxide [16,17].

Cocos nucifera is a well-known plant which is a great resource of vitamins, minerals, proteins, carbohydrates and tannins etc. It has industrial and medicinal applications and its parts can be used as antibacterial, anti-inflammatory, antiviral agent in many fields [18-21]. Elango *et al.* have prepared Lead nanoparticles via the agricultural waste of *Cocos nucifera* plant [22]. Roopan *et al.* have reported the green synthesis of Pd NPs [23], Ni-Pd nanoparticles [24], Silver nanoparticles [25] and Nickel nanoparticles [26] from the extract of *Cocos nucifera* coir and studied their biological activities.

In the present work, immature fruit extract of *Cocos nucifera* was used for the preparation of nanoparticles. The immature coconut fruit is a biowaste which falls from the coconut tree due to pest damage, temperature changes, plant diseases, summer drop, drought stress/pruning or other unfavourable conditions.

The bio synthesized nanoparticles from plant extracts can be used as catalysts for various organic transformation reactions. Very few reports are available in the literature, for the biotransformation using biosynthesized nanoparticles. Edison *et al.* have reported the reduction of ortho and para nitroaniline by sodium borohydride with silver nanoparticles synthesized by the aqueous extract of *Tamarindus indica* (T. indica) seed coat [27]. Raghasudha have synthesized silver nanoparticles from the leaf extract of *Syzigium cumini* and used the synthesized nanoparticles as catalyst for the reduction of p-Nitrophenol to p-aminophenol [28]. Aswathy Aromal *et al.* have reported the green synthesis of gold nanoparticles using the aqueous extract of fenugreek (*Trigonella foenum-graecum*). They used the synthesized Au nanoparticles as catalyst for the reduction of nitrophenol to amino phenol and studied their size dependent catalytic activity [29].

In the present work, we have synthesized three metal oxide nanoparticles (CuO, NiO and Fe₂O₃) from the aqueous extract of immature fruit of *Cocos nucifera* and the metal oxides were characterized using UV-Vis, FT-IR, XRD, SEM, EDAX and TEM analytical procedures. The synthesized MO nanoparticles were used as catalysts for the reduction of aromatic aldehydes.

2 Materials and Methods

The fresh immature coconut fruit which was fallen from the coconut tree was collected from Palayamkottai area, Tirunelveli, Tamil Nadu, India. Nickel (II) Chloride hexahydrate, Ferric Chloride, p-Chlorobenzaldehyde and Sodium hydroxide were purchased from SRL chemicals. Copper Sulphate penta hydrate (CuSO₄.5H₂O) was purchased from Nice chemicals, India. Ammonium formate was purchased from Loba chemicals. All the reagents used were of AR Grade. All aqueous solutions were made up of double distilled water.

2.1 Synthesis of metal oxide nanoparticles

The flesh of the immature coconut fruit was cut into small pieces with an ethanol sterilized knife and ground with a mortar and pestle. Then it was dispensed in 100 ml of distilled water for 5 minutes. The extract was filtered using Whatman no. 1 filter paper (Fig. 1 a).

About 100 ml of the immature coconut fruit extract was mixed with 100 ml of 0.1 M of aqueous Nickel (II) chloride hexahydrate/ Copper sulphate penta hydrate/ Ferric Chloride salt solution separately under stirring condition (Fig. 1 b). Fe₂O₃ nanoparticles was settled down immediately as brown colour precipitate. However, NiO and Cuprous Oxide nanoparticles were formed after the addition of 5 ml of 0.1 M sodium hydroxide solution into the reaction mixture separately (Fig. 1 c). The precipitate was then filtered and dried at 100°C for 2 h in a hot air oven.

2.2 Characterization of Metal Oxide Nanoparticles

Optical absorption spectra of the synthesized NPs were taken with the UV-Vis Double Beam spectrophotometer 2201, Systronics using a quartz cuvette of 1 cm path length. FT-IR spectra of the nanoparticles prepared were recorded at room temperature on IR affinity (FT-IR spectrophotometer, Shimadzu) ranging from 4000 to 400 cm⁻¹. The morphology of the synthesized nanoparticle was studied by Scanning Electron Microscopy (Jeol, Karunya University, India). The synthesized MO nanoparticles were analysed by powder X-Ray diffraction technique using X-Ray Diffractometer (XPERT-PRO, Alagappa university, India) with the copper $K_{\alpha 1}$ radiation (k = 1.54 A) in the 2h range of 10° – 80°. The elemental composition and stoichiometry of the synthesized nanoparticles was studied by EDAX instrument (Oxford Instruments, Karunya University, India). TEM analysis of Nickel Oxide nanoparticles were done at SAIF, Cochin, India.

2.3 Catalytic activity of nanoparticles on the reduction of aromatic aldehydes

Aromatic aldehyde (30 mg, 0.2 mmol) dissolved in 2 ml of ethanol was mixed with the MO nanoparticles as catalyst (1 mg, 1:30 ratio). Ammonium formate (0.3 mmol, 1.5 equivalent) and sodium hydroxide (0.15 mmol) dissolved in water were added to the above solution and heated to 60 °C. The reaction was monitored by TLC. After completion of the reaction, the catalyst was filtered and the organic layer was evaporated. The crude product was dissolved in ethyl acetate and washed with sodium bisulphite solution to remove the unreacted aldehyde. The crude products were purified by column chromatography.

3 RESULTS AND DISCUSSION

The biowaste, immature coconut fruit, was chosen to prepare the metal oxide nanoparticles. Phytochemical analysis (Table.1) was done to identify the presence of various phytoconstituents in the aqueous extract of immature fruit of *Cocos nucifera*. Phytoconstituents such as alkaloids, steroids, reducing sugars, carbohydrates, phenolic compounds, flavonoids, saponins and tannins were present in the immature coconut fruit extract.

NiO, Cu₂O and Fe₂O₃ nanoparticles were synthesized using the aqueous immature fruit extract of *Cocos nucifera*. Fe₂O₃ nanoparticles were formed at neutral pH whereas NiO and Cu₂O nanoparticles were formed at basic pH. Further, the synthesized nanoparticles were characterized by spectral techniques.

3.1 Characterization of the synthesized nanoparticles

3.1.1 UV-Vis spectra

UV-Vis spectrum of NiO nanoparticles (Fig. 2 (a)) showed a broad absorption peak at 333 nm corresponds to the formation of nanoparticles [30-32]. UV-Vis spectrum of Fe₂O₃ nanoparticles in Fig. 2 (b) showed absorption band at 201 nm confirming the formation of Fe₂O₃ nanoparticles [33]. Cuprous Oxide (Cu₂O) nanoparticles (Fig 2 (c)) showed absorption band in the range of 398 nm - 569 nm. Absorption at ~410 nm indicates the formation of Cu₂O

nanoparticles [34] and the two small bands at 484 nm and 560 nm are due to the phytoconstituents present in the plant extract.

3.1.2 FT-IR spectra of Metal Oxide nanoparticles

Fig. 3 showed the FT-IR spectra of the synthesized metal oxide nanoparticles. In the FT-IR spectrum of Metal Oxide (MO) nanoparticles, absorption peaks around 3300 cm⁻¹, 1600 cm⁻¹ and 1000 cm⁻¹ correspond to H-O-H stretching, H- bonded alcohols and phenols, C-H alkyl stretching respectively. It confirmed the presence of phytoconstituents like polyphenols, alkaloids, steroids which were responsible for the reduction of metal precursors into Metal Oxide nanoparticles. The IR band at 455 cm⁻¹ in the FT-IR spectrum of NiO nanoparticles confirmed the Ni-O stretching vibration. The strong absorption band at 516 cm⁻¹ in the IR spectrum of iron oxide was due to the Fe-O stretching vibration. In the FT-IR spectrum of Cuprous Oxide nanoparticles, the peaks at 470.63 cm⁻¹ (Cu-O symmetric stretching),) and 601.79 cm⁻¹ (Cu-O wagging) implied the presence of metal-oxide group in the sample [35].

3.1.3 X-Ray Diffraction (XRD) analysis

Fig. 4 denotes the XRD spectra of the synthesized MO nanoparticles. Peaks at 20 values of 38.2° , 44.4° , 77.7° in the XRD spectrum of Nickel Oxide nanoparticles (JCPDS: 47-1049) confirmed that the structure was face centred cubic [30]. XRD spectrum of Fe₂O₃ nanoparticles was broad indicating that the particles fell in nanometre range. Cuprous oxide nanoparticles showed diffraction patterns at 35.2° , 38.4° , 48.4° which were related to the single phase monoclinic (JCPDS: 80-1916) structure. The particles size was calculated using Debye-Scherrer formula using the FWHM of the most intense peak. The size of the NiO, Fe₂O₃ and Cu₂O nanoparticles were of 24 nm, 53 nm and 51 nm respectively.

The formation of NiO nanoparticles was further confirmed by comparing the 2θ values in the XRD spectrum of biosynthesized NiO nanoparticles with that of chemically synthesized NiO nanoparticles (Fig. 4 (b)).

3.1.4 SEM analysis

Surface morphology of the synthesized metal oxide nanoparticles was studied by SEM analysis. SEM image of NiO nanoparticles (Fig. 5 (a)) confirmed that the particles were highly mono dispersed and have deformed cubic size. Fig. 5 (b) implied that Cuprous Oxide nanoparticles were spherical in shape. SEM image of Fe₂O₃ nanoparticles in Fig. 5 (c) showed that the particles were in distorted spherical shape surrounded by the stabilizing agents from the plant extract. Since, Fe₂O₃ nanoparticles were formed in neutral medium in contrast to the NiO and Cu₂O NPs, the stabilizing agents are tangible in the SEM image of iron oxide nanoparticles.

3.1.5 EDAX analysis

The elemental composition of the synthesized nanoparticles was analysed using EDAX spectrum. Table. 2 presents the composition of metal oxide nanoparticles. EDAX spectrum (Fig. 6 (a)) confirms the formation of NiO nanoparticles. The presence of Si [36] and K were originated from the biomolecules of the plant extract. EDAX spectrum (Fig. 6 (c)) confirms the formation of Cu₂O (Cuprous Oxide) nanoparticles. EDAX spectrum of iron oxide nanoparticles strongly confirms the formation of Fe₂O₃ nanoparticles (Fig. 6 (b)).

3.1.6 TEM analysis

TEM and SAED pattern of Nickel Oxide nanoparticles are shown in Fig. 7. The SAED pattern of NiO nanoparticles confirms the nanoparticle formation and the exact size of the NiO nanoparticles was 51 nm.

3.2 Reduction of aromatic aldehydes

The synthesized nanoparticles were used for the reduction of aromatic aldehydes. p-Chlorobenzaldehyde was taken as the model substrate. The reduction was carried out using ammonium formate as the hydrogen donor in basic pH at 60 °C. Among the nanoparticles used, NiO showed high catalytic activity as evident from the high yield of the p-Chlorobenzyl alcohol (90%) in 2 h (Scheme. 1). This was mainly due to the large surface area and high activity of NiO nanoparticles as heterogeneous catalyst than the other MO nanoparticles. Cu₂O and Fe₂O₃ nanoparticles gave the alcohol in 32% and 20% yield respectively. When the reaction was done without the catalyst, there was no conversion of the product in 2 h. When the reaction was carried out at room temperature, the yield was only 57%.

Since NiO nanoparticles has shown remarkable catalytic activity, it was used for the subsequent optimization process. Further, the effect of solvent on the reduction of p-Chlorobenzaldehyde was studied using various hydrophilic solvents like ethanol, methanol, tetrahydrofuran, acetonitrile and a mixture of water and methanol. Among the solvents used, ethanol was found to be the best solvent to reduce the aldehyde to a higher extent (Scheme. 2).

Further, when the reduction was carried out by varying the amount of catalyst loading, it was observed that increase in the amount of catalyst did not alter the yield of the reaction (Fig. 8).

Then, the reaction was optimized by using various nickel catalysts synthesized by various methods (Table. 3). Montmorillonite clay supported Nickel nanoparticles [37] gave the reduced product in low yield (34%). Cysteine capped Nickel nanoparticles [38] gave the alcohol in moderate yield (58%). When the reduction was done using NiCl₂ salt as catalyst, the reaction did not proceed. NiO nanoparticles prepared from immature fruit of *Cocos nucifera* has only shown promising catalytic activity.

3.2.5 Reduction of benzaldehyde substrates

Having optimized the reaction conditions for the reduction of p-Chlorobenzaldehyde, various aromatic aldehydes were reduced using NiO nanoparticles (Scheme. 3). The electronic nature of the substituents in the benzene ring did not affect the yield of the reaction. The electron donating methoxy and methyl substituents and electron withdrawing nitro group did not make a pronounced effect on the course of the reaction and the products were obtained in good yields (Table. 4). However, the reaction time varied for each substrate (2-24 h). p-Bromobenzaldehyde, p-Chlorobenzaldehyde and 2-Hydroxybenzaldehyde were reduced in 83%, 90% and 81% yield respectively.

Conclusion

In summary, metal oxide nanoparticles were synthesized using the aqueous immature fruit extract of *Cocos nucifera* in a green synthetic route. The catalytic activity of the synthesized nanoparticles was examined for the reduction of aromatic benzaldehydes. It was found that Nickel Oxide nanoparticles showed excellent yields compared to the other synthesized nanoparticles. The reduction was carried out without any harsh conditions using ammonium formate as green hydrogen source.

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Figure Captions

Fig. 1. Synthesis of metal oxide nanoparticles (a) Aqueous immature coconut fruit extract (b) Addition of metal salt into extract (i) CuSO₄. 5H₂O (ii) FeCl₃ (iii) NiCl₂. 6H₂O (c) Formation of Metal Oxide nanoparticles



Fig. 2. UV-Visible spectra of (a) NiO nanoparticles (b) Fe_2O_3 nanoparticles (c) Cu_2O nanoparticles





Fig. 3. Fourier-transform infrared spectra of (a) NiO nanoparticles (b) Fe₂O₃ nanoparticles (c) Cu₂O nanoparticles



Fig. 4. (a) Powder X-Ray Diffraction spectra of (i) NiO nanoparticles (ii) Fe₂O₃ nanoparticles (iii) Cu₂O nanoparticles



Fig. 4 (b) X-Ray diffraction spectra of biosynthesized and chemically prepared NiO nanoparticles



Fig. 5. Scanning Electron Microscopic images of (a) NiO nanoparticles (b) Cu₂O nanoparticles

(c) Fe₂O₃ nanoparticles







Fig. 6. Energy dispersive analysis x-ray spectra of (a) NiO nanoparticles (b) Fe_2O_3 nanoparticles (c) Cu_2O nanoparticles





Fig. 7. Transmission Electron Microscopic images of NiO nanoparticles at different magnifications (a) 50 nm (b) 20 nm (c) Selected area electron diffraction image of NiO nanoparticles



Fig. 8. Reduction of p-Chlorobenzaldehyde by varying catalyst loading



Supporting Information

(i) Benzyl alcohol

Colourless liquid. **FT-IR Data**: 3606 cm⁻¹, 3204 cm⁻¹, 2167.84 cm⁻¹, 1640.35 cm⁻¹, 1426.26 cm⁻¹ (Fig. 1). ¹**H NMR** (400 MHz, CDCl₃) (Fig. 2): 2.8 ppm (s, 1H, -OH), 4.6 ppm (s, 2H, -CH₂-) and 7.24-7.33 ppm (m, Ar-H). ¹³**C NMR**: 64.84 ppm-65.08 ppm, 115.46 ppm, 120.06 ppm, 126.70 ppm - 129.57 ppm, 140.61 ppm - 140.91 ppm and 156.39 ppm (Fig. 3).



Fig. 1 FT-IR spectrum of Benzyl alcohol



Fig. 2 ¹H NMR spectrum of Benzyl alcohol



Fig. 3 ¹³C NMR spectrum of Benzyl alcohol

(ii) 4-Methoxybenzyl alcohol

Colourless liquid. **FT-IR data**: 3356.87 cm⁻¹, 3002 67 cm⁻¹, 2936.42 cm⁻¹, 1462.91 cm⁻¹ ¹ and 1586.34 cm⁻¹ (Fig. 4). ¹**H NMR** (400 MHz, CDCl₃) (Fig. 5): 3.73 ppm (s, 3H, -CH₃), 4.47 ppm (s, 2H, -CH₂-), 3.4 ppm (s, 1H, -OH), 6,81-6.83 ppm (d, Ar-H) and 7.18-7.20 ppm (d, Ar-H). ¹³**C NMR**: 159.23 ppm, 133.13 ppm, 128.67 ppm, 113.97 ppm, 65.06 ppm and 55.32 ppm (Fig. 6).



Fig. 4 FT-IR spectrum of 4-Methoxybenzyl alcohol



Fig. 5 ¹H NMR spectrum of 4-Methoxybenzyl alcohol



Fig. 6 ¹³C NMR spectrum of 4-Methoxybenzyl alcohol

(iii) p-Nitrobenzyl alcohol

Straw yellow solid. Melting point: 92°C. **FT-IR data**: 3518.88 cm⁻¹, 3096.75 cm⁻¹, 2866.99 cm⁻¹, 1508.23 cm⁻¹ and 1604.81 cm⁻¹, 1338.51 cm⁻¹, 1056.92 cm⁻¹, 1458.08 cm⁻¹ (Fig. 7). ¹**H NMR** (400 MHz, CDCl₃) (Fig. 8): 7.52-7.54 ppm (d, Ar-H), 8.18-8.21 ppm (m, Ar-H), 4.82-4.83 ppm (d, 2H, -CH₂) and 2.22 ppm (s, 1H, -OH). ¹³C **NMR**: 63.99 ppm, 123.73 ppm, 127.01 ppm, 127.30 ppm, 147.26 ppm and 148.24 ppm (Fig. 9).



Fig. 7 FT-IR spectrum of p-Nitrobenzyl alcohol



Fig. 8 ¹H NMR spectrum of 4-Nitrobenzyl alcohol



Fig. 9¹³C NMR spectrum of 4-Nitrobenzyl alcohol

(iv) p-Chlorobenzyl alcohol



Fig. 10 FT-IR spectrum of p-Chlorobenzyl alcohol



Fig. 11 ¹H NMR spectrum of p-Chlorobenzyl alcohol



Fig. 12¹³H NMR spectrum of p-Chlorobenzyl alcohol

White solid. Melting point: 70 °C. **FT-IR data:** 3169 cm⁻¹, 3032 cm⁻¹, 2874 cm⁻¹, 1602 cm⁻¹, 1496 cm⁻¹ and 1454 cm⁻¹ (Fig. 10). ¹**H NMR** (400 MHz, CDCl₃) (Fig. 11): 2.01 ppm (s, 1H, -OH), 4.63 ppm (s, 2H, -CH₂-) and 7.26-7.32 ppm (m, Ar-H). ¹³**C NMR**: 64.51 ppm, 128.28-128.68 ppm, 133.35 ppm and 139.25 ppm (Fig. 12).

(v) p-Bromobenzyl alcohol

White solid. Melting point: 76 °C. **FT-IR data**: 3221.87 cm⁻¹, 2917 cm⁻¹, 2718.48 cm⁻¹, 1658 cm⁻¹, 1485 cm⁻¹ and 1446 cm⁻¹ (Fig. 13). ¹**H NMR** (400 MHz, CDCl₃) (Fig. 14): 2.8 ppm (s, 1H, -OH), 4.65 ppm (s, 2H, -CH₂-), 7.23-7.25 ppm (d, Ar-H) and 7.47-7.49 ppm (d, Ar-H). ¹³C NMR: 64.41-64.45 ppm, 121.41 ppm, 128.29-128.87 ppm, 131.34 -131.89 ppm and 139.79 ppm (Fig. 15).



Fig. 13 FT-IR spectrum of 4-Bromobenzyl alcohol



Fig. 14 ¹H NMR spectrum of 4-Bromobenzyl alcohol



Fig. 15¹³C NMR spectrum of 4-Bromobenzyl alcohol

(vi) p-Methylbenzyl alcohol

White solid. Melting point: 61°C. **FT-IR data:** 3259.47 cm⁻¹, 3020.32 cm⁻¹, 2920.03 cm⁻¹, 1614 cm⁻¹, 1443.62 cm⁻¹ and 1586.34 cm⁻¹ (Fig. 16). ¹H NMR (400 MHz, CDCl₃) (Fig. 17): 2.34 ppm (s, 3H, -CH₃),3.65 ppm (s, 1H, -OH), 4.6 ppm (s, 2H, -CH₂-), 7.14-7.16 ppm (d, Ar-H), 7.22-7.24 ppm (d, Ar-H). ¹³C NMR: 21.18 ppm, 64.95-65.01 ppm, 126.85–127.42 ppm, 128.95-129.47 ppm, 137.27 ppm and 137.70-138 ppm (Fig. 18).



Fig. 16 FT-IR spectrum of 4-Methylbenzyl alcohol



Fig. 17 ¹H NMR spectrum of 4-Methylbenzyl alcohol



Fig. 18¹³C NMR spectrum of 4-Methylbenzyl alcohol

(vii) 2-Hydroxybenzyl alcohol

White solid. Melting point: 85 °C. **FT-IR data:** 3735.86 cm⁻¹, 3461.02 cm⁻¹, 2854.45 cm⁻¹, 1491.84 cm⁻¹ and 1587.31 cm⁻¹ (Fig. 19). ¹**H NMR** (400 MHz, CDCl₃) (Fig. 20): 3.7 ppm (s, 1H, -OH), 4.6 ppm (s, 2H, -CH₂), 5.35 ppm (s, 1H, -OH), 6.7 ppm (d, 1H, -CH), 6.9 ppm (d, 1H, -CH), 7.24 ppm (d, 1H, -CH) and 7.27 ppm (d, 1H, -CH). ¹³C NMR: 61 ppm, 117 ppm, 124.3 ppm, 128.75 ppm, 129.8 ppm, 130.1 ppm and 156.2 ppm (Fig. 21).



Fig. 19 FT-IR spectrum of 2-Hydroxybenzyl alcohol



Fig. 20¹H spectrum of 2-Hydroxybenzyl alcohol



Fig. 21 ¹³C spectrum of 2-Hydroxybenzyl alcohol



Scheme 1. Reduction of p-Chlorobenzaldehyde using various metal oxide nanoparticles



1	Nickel Oxide	90
2	Cuprous Oxide	32
3	Fe ₂ O ₃	20
4	No catalyst	~ ~

Scheme. 2 Reduction of p-Chlorobenzaldehyde in different solvents



Scheme 3. Reduction of benzaldehyde substrates using NiO nanoparticles



R = - H, - OCH₃, - NO₂, - Br, - Cl, - CH₃, - OH

No.	Experiment	Observation	Interference
1	Steroids:	Purple color changed to	Presence of
1.	Libermann-Burchard reagent	blue color	steroids
2.	Reducing sugar : (i) Fehlings' solution A and B	Reddish brown precipitate	Presence of reducing sugar
	(ii)Tollens' reagent	Silver mirror	5 5
3.	Alkaloids: Mayer's reagent	White turbidity	Presence of Alkaloids
4.	Phenolic compounds: FeCl ₃ test	Intense color	Presence of phenolic compounds
5.	Carbohydrates: Molisch's reagent	Purple color	Presence of Carbohydrates
6.	Saponins: Emulsion Test	Emulsion formed	Presence of Saponins
7.	Xanthoproteins: Con. HNO ₃	No reddish orange precipitate	Absence of Xanthoprotein
8.	Coumarins: Chloroform + NaOH	No yellow color	Absence of Coumarins
9.	Flavonoids: Con. HCl	Orange color	Presence of Flavonoids
10.	Tannins: Lead acetate + NaOH	White precipitate	Presence of Tannins

Table.1 Phytochemical analysis of aqueous immature fruit extract of Cocos nucifera

Table: 2 Elemental composition of metal oxide nanoparticles observed from Energy

Elements	Composition	Elements	Composition	Elements in	Composition
in NiO		in Cu ₂ O		Fe_2O_3	
Ni	37.13	Cu	52.97	Fe	41.04
Si	1.88	Si	3.98	0	58.32
0	60.99	0	43.05	Si	0.49
				К	0.15
Total	100		100		100

Dispersive Analysis X-ray spectra

S. No.	Catalyst	Yield (%)
1	NiO from Cocos nucifera	90
2	Ni-MMT	33.8
3	Ni-Cys	58
4	NiCl ₂ salt	No reaction

Table: 3 Reduction of p-Chlorobenzaldehyde by various Nickel catalysts

Table. 4 Reduction of benzaldehyde substrates using NiO nanoparticles

S. No.	Reactant	Product	Time	Yield ^a (%)
1,	СНО	Н2С-ОН	2 h	89
2.	0	HO	4 h	82
3.			4 h	87



^a Reaction condition: Reactant aldehyde (0.2 mmol), ammonium formate (0.3 mmol), sodium hydroxide (0.15 mmol) and NiO nanoparticles (1 mg).