Microwave-assisted synthesis of cobalt oxide nanoparticles with *Carica* papaya leaf extract and their catalytic activities

ABSTRACT

The Physico-chemical and optical characteristics of cobalt oxide nanoparticles (CoONPs) have attracted interest in their creation over the past few years. In the current study, microwave irradiation was used to induce the production of CoONPs from papaya leaf extract. Papaya leaf extract was prepared by pulverizing and drying the plant's leaves. The extract was then heated to create an aqueous extract, which was purified and saved for analysis. Cobalt nitrate was added to the extract, and a dark brown color was obtained. CoONPs were used as a catalyst in the reduction of MB and CR, with a mixture of MB solution and NaBH4 in DD water. The catalytic reduction of 4-NP was also tested using CoONPs. The experimental procedures for characterizing *Carica*papaya leaf extract CoO NPs include UV-Vis spectra, TEM, and FTIR for analyzing nanomaterial morphology, size, and chemical composition. X-ray Diffraction (XRD) spectroscopy provides information about the crystalline structure, which is crucial for understanding nanomaterial behavior and potential applications in fields like medicine, electronics, and materials science. These methods are critical for understanding nanomaterial behavior and potential applications in various fields.

Keywords: *Carica* papaya leaf extract, CoONPs, FTIR, TEM, XRD, Catalytic activities

1. INTRODUCTION

Despite the fact that contemporary enterprises have expanded rapidly over the past few decades, environmental degradation has so far become a severe threat to human well-being on a global scale. The contamination of water bodies caused by the discharge of untreated water containing inorganic and organic species has received a lot of attention recently among the numerous types of environmental pollution[1]. Aquatic habitats are thought to suffer severe harm from the unregulated release of azo dyes, which are frequently employed in the leather, textile, plastic, cosmetics, ink, paper, and food sectors[2]. Organic dyes can last for a long time in the environment because of their photo- and thermal stability, as well as their capacity to withstand biodegradation[3,27,28,29]. According to reports, the presence and persistence of these oxygen-sequestering species in water systems reduces light penetration and prevents aquatic vegetation from photo synthesizing. Therefore, before industrial effluents containing hazardous organic dyes may be properly discharged to the environment, they must be broken down and decolored.

The U.S. EPA classifies phenolic chemicals, including the well-known nitrophenol derivatives, along with organic azo dyes as priority pollutants that have a direct impact on human health and the environment[4].

Up to date, a wide range of technologies have been created to remove colorants from waste water, including physical absorption, chemical oxidation, chemical degradation, membrane filtration, biological degradation, and membrane filtering[5]. Due to their exceptional catalytic activity and effective color removal, metal oxide nanoparticles have received the most interest when it comes to the catalytic degradation of organic dyes[6]. However, a number of obstacles prevent the practical application of these nanoparticle catalysts. For instance, recycling these colloidal nanoparticles in an aqueous solution for environmentally friendly purposes is challenging[7]. Additionally, the nanoparticles tend to agglomerate due to their high surface energy and huge surface area. These days, a variety of materials, including graphene, Fe_3O_4 microspheres, zeolites, polymeric microspheres, etc., have been used as supports[8].The nanoparticles, which have strong antibacterial properties, are valuable in the pharmaceutical industry due to their antibacterial properties [26].

Due to their unique characteristics, cobaltoxide nanoparticles (CoONPs) have received a lot of attention. Gas sensors, catalysis, batteries, high temperature superconductors, and solar energy conversion devices are just a few of the uses for CoONPs. CoONPs are much more durable, stable, and long-lasting than organic anti-microbial compounds[9]. CoONPs can be made using a variety of physical and chemical processes. However, these techniques have limitations, including the use of hazardous reducing agents, organic solvents, and high temperatures and pressures[10]. The green synthesis of nanoparticles using plant extracts, however, holds great promise for increasing nanoparticle production without the use of hazardous or expensive chemicals[11]. Therefore, it is necessary to create and apply safe synthetic procedures that are economical, efficient, nontoxic, and friendly to the environment. Recently, it was reported that Populus ciliata leaf extract was used in the manufacture of CoONPs[12].

In the present study, we are reporting the synthesis of CoONPs using papaya leaf extract. Papaya belongs to the family Caricaceae and is a medicinal plant[13]. Numerous active substances, including papain, chymopapain, cyanogenic glucosides, cystatin, -tocopherol, ascorbic acid, flavonoids, and gluco-sinolates, have been found to be present in papaya leaves[14]. Several illnesses, including amenorrhea, general debility, constipation, corns, cutaneous tubercles, warts,

sinusitis, eczema, glandular tumors, blood pressure, diabetes, malaria, intestinal worms, and syphilis are treated with papaya bark, leaves, and fruits[15].

In the current study, a microwave irradiation technique has been developed to produce CoO NPs using papaya leaf extract, a cheap and abundant natural resource and Synthesized CoO NPs have been characterized using UV-Vis, FTIR, XRD, and TEM. The resulting CoO NPs are spherical in shape and highly crystalline. When containing NaBH4, these capped CoO NPs are effective in reducing 4-NP, MB, and CR compounds. This innovative catalyst is expected to be widely used in organic catalysis, making it suitable for treating industrial effluents containing toxic contaminants.

2. MATERIALS AND METHODS

2.1. Chemicals and reagents

Cobalt nitrate hexahydrate ($Co(NO_3)_2.6H_2O$), (99%), was bought from Sigma Aldrich, India. The 4-NP, MB, and CR dyes were procured from Merck, India The other substances employed in this experiment were all analytic reagent grade and didn't require any additional purification. All of the investigations were conducted using double distilled (DD) water.

2.2. Preparation of papaya leaf extract

The papaya plant's fresh leaves were plucked, finely chopped, and allowed to dry in the shade. It was pulverized and used for further research after drying. 2 grams of leaf extract powder were added to a 100 ml Erlenmeyer flask containing double-distilled water and heated for 30 minutes at 55 °C to create the aqueous extract. The resulting extract was purified with Whatman No. 1 filter paper, and the filtrate was saved for later analysis.

2.3. Synthesis of CoONPs

A source of cobalt, 50 ml of cobalt nitrate with a 2 mM concentration, was added dropwise into 15 ml of extract, which was then continuously stirred at room temperature for 30 min. A microwave irradiation at 450W power for 4 minutes followed, and the resulting dark brown color indicated that CoONPs were successfully synthesized[9].

2.4. Catalytic reduction of MB and CR

In the presence of $NaBH_4$, CoONPs were used as a catalyst in the reduction of MB and CR. In a typical procedure, 2.5 mL of 1 mM MB solution was mixed with 1.5 mL of 10 mM $NaBH_4$ in DD water and stirred for 5 minutes. In a cuvette, 3 mL of this reaction mixture was added to 10

mg of CoONPs, and UV-Vis spectra were recorded at various time intervals. In addition, CR dye was used in the same way[1].

2.5. Catalytic reduction of 4-NP

CoONPs were tested for their catalytic activity by reducing 4-NP. In a typical experiment, 1.6 ml of 4-NP (0.2M) and 1 ml of $NaBH_4$ (2 mM) were combined in an aqueous solution and placed in a quartz cuvette for the reaction. The aforementioned combination received 10 mg of CoONPs, and the reaction was watched by capturing the UV-Vis spectra at 1-minute intervals[2].

2.6. Characterization of *Carica* papaya leaf extract CoONPs

The experimental procedures for characterizing *Carica* papaya leaf extract CoO NPs include a number of sophisticated techniques for measuring their unique characteristics. UV-Vis spectra (UV-Vis, Shimadzu, spectrum one), Transmission Electron Microscopy (TEM, JEOL), and Fourier Transform Infrared Spectroscopy (FTIR, Agilent) are common techniques for analyzing nanomaterial morphology, size, and chemical composition. Additionally, X-ray Diffraction (XRD, Rigaku) spectroscopy provides information about the crystalline structure. These methods are critical for understanding nanomaterial behavior and potential applications in a range of fields, such as medicine, electronics, and materials science.

3. RESULTS AND DISCUSSION

3.1. UV-Visible spectra

UV-visible spectra were used to examine the progression of the reaction between the components in the extract and the cobalt ions. The UV-Visible absorption spectrum of synthesized CoONPs was showed in **Figure.1**, the peak appeared at 504 nm. This absorption band is the source of the CoONPs plasma resonance absorption. The continuous oscillation of the electrons in the conduction band brought on by the interaction of the electro-magnetic field is the cause of light absorption by nanoparticles. The surface plasmon absorption band that the CoONPs display in the range of 350–550 nm is their distinctive property[16]. The extracts function as a reducing-cum-surface capping agent, which is responsible for the synthesis of CoONPs.

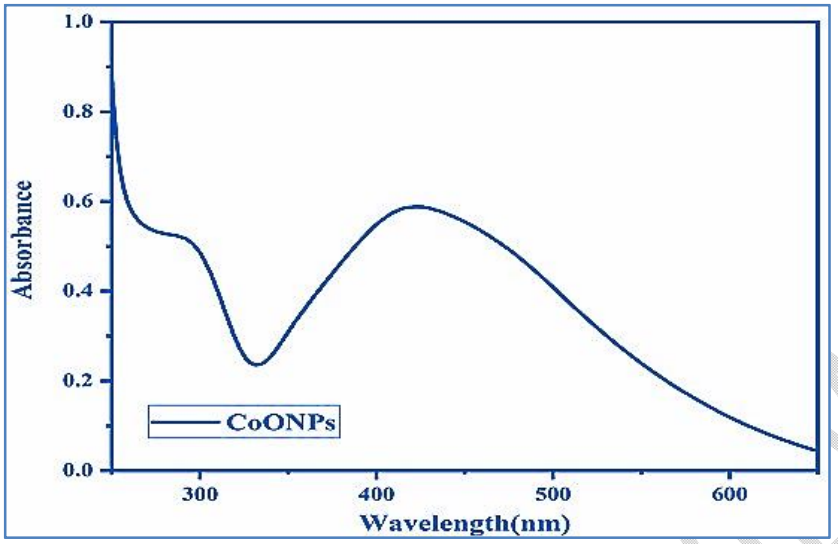

Figure.1 UV-Vis spectra of CoONPsprepared using papaya leaf extract

3.2. FTIR

The potential biomolecules responsible for CoONPs stabilization and reduction were determined using FTIR measurements. The FTIR spectrum of the extract mediated synthesized CoONPs shown in **Figure. 2**. The presence of phenol functionalities in papaya leaf extract is revealed by FT-IR analysis. The phenol O-H stretching groups are represented as absorption peaks in the spectrum at about 3343 cm^{-1}, 1740 cm^{-1} peak is corresponds to carbonyl group, 1608 cm^{-1} from the resonance groups of the aromatic C=C, and 2917 cm^{-1} from the C-H aliphatic. C-H deformations and in-plane O-H bending was assigned to absorptions at 1352 cm^{-1}. Peaks from 1022 cm^{-1} were found to correlate to C-O-C glycosidic linkage vibrations as well as secondary alcohols and phenols C-O stretching and O-H deformation vibrations. As a result of the characteristic peaks observed in the FTIR spectrum of the as-synthesized CoONPs, flavonoids, steroids, saponins, tannins, alkaloids, phenols, sugar, saccharides, and proteins were also found in the papaya extract. This confirms that these chemicals can also act as reducing agents as well as cappers[17].

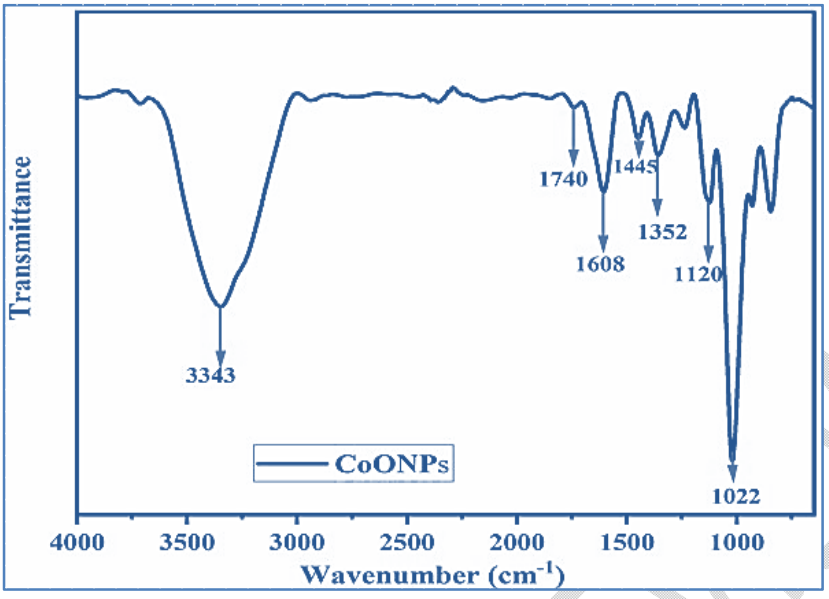

Figure.2 FTIR spectra of synthesized CoONPs

3.3. XRD

The XRD analysis was evaluated the phase and crystal structure of the synthesized CoONPs. The characteristic peaks in the green synthesized CoONPs XRD pattern at 31.66, 36.45, 45.48, 56.53, 61.45, 66.17 and 75.25, which were corresponds to 220, 311, 400, 422, 511, 440 and 533 respectively **(Figure.3).**The International Centre for Diffraction Data card no. 42-1467 indicates the formation of the crystalline phase of CoONPs[18]. CoONPs produced by various plant extracts have been reported to have similar XRD patterns.

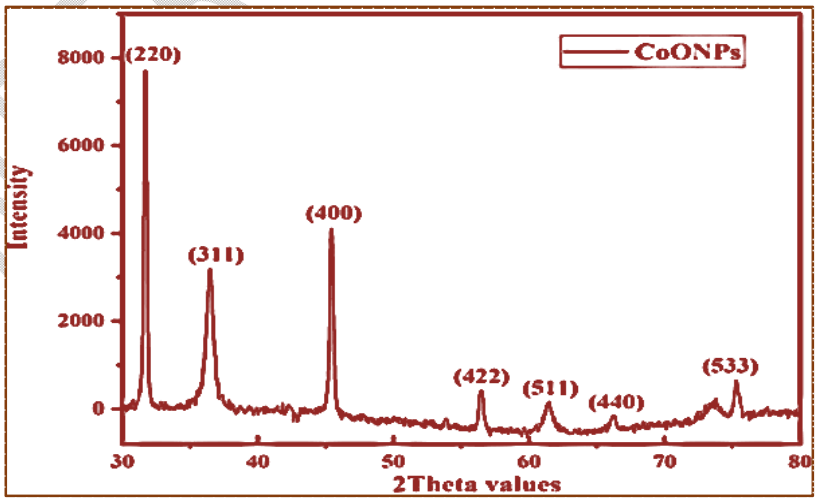

Figure.3 XRD pattern of synthesized CoONPs

3.4. TEM

The TEM examination was utilized to know the size, shape, morphology of CoONPs were and TEM image of CoONPs are displayed in **Figure 4 (a).** This makes it clear that the majority of the nanoparticles are spherical in shape and evenly distributed. The particle size distribution histogram makes it evident that the size of the generated nanoparticle is between 3 and 20 nm **(Figure 4b).** It is discovered that the average particle size is 8±2 nm.

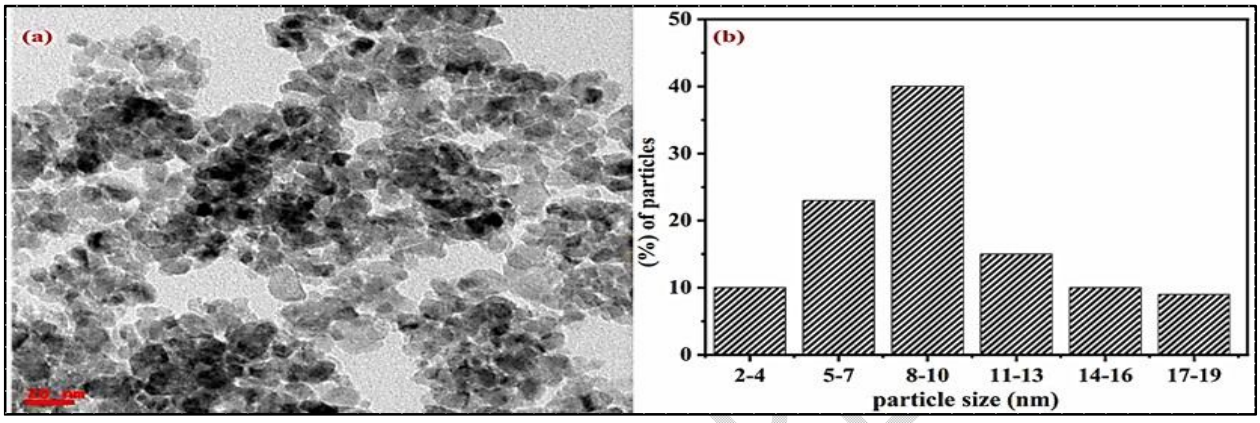

Figure.4(a) TEM image of synthesized CoONPs and (b) particle size distribution of CoONPs

Catalytic activity of CoONPs

Numerous studies have examined the catalytic activity of CoONPs in the reduction of various dyes, including 4-NP, MB, and CR, and biosynthesized CoONPs have been shown to be a very effective catalytic element due to their simple, affordable, and environmentally friendly synthesis process. The reducing agent $NaBH_4$ was frequently used as a model reaction while examining these nanoparticles' catalytic potential.

3.5. Catalytic reduction of MB

In the case of MB, it is a dye that is frequently used in biology and chemistry, can build up, and may be harmful to the environment. When reduced, MB loses its blue and turns into the colorless (leuco methylene blue). Figure 5a depicts the UV-Vis absorption spectra of the reduction of MB by $NaBH_4$ in the absence of the catalyst CoONPs. Even after 30 minutes, the magnitude of the MB absorption peak decreases slightly when $NaBH_4$ is present in **Figure 5a**. These findings suggest that in the absence of CoONPs, the reduction reaction was slow. As can be seen in **Figure 5b**, the UV-Vis spectrum of the $NaBH_4$ reduction of MB in the presence of CoONPs catalysts was obtained[19]. Due to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions, MB normally exhibits an

absorption maximum band at about 664 nm in aqueous solution. But when CoONPs were added to the aforementioned reaction mixture, MB was completely reduced in just 7min. According to Fig. 5b, the MB has reduced as seen by the continuous decrease in MB's absorption peak strength at 664 nm with increasing time. Furthermore, the procedure never reached its peak appearance. At 10 mg of CoONPs and room temperature, the reduction of MB follows a pseudo-first order kinetics, as shown by the linear relationship between $\ln(A_t/A_0)$ vs reaction time **(Figure 5c)**[1]. The rate constant was determined to be 0.332 min^{-1}.

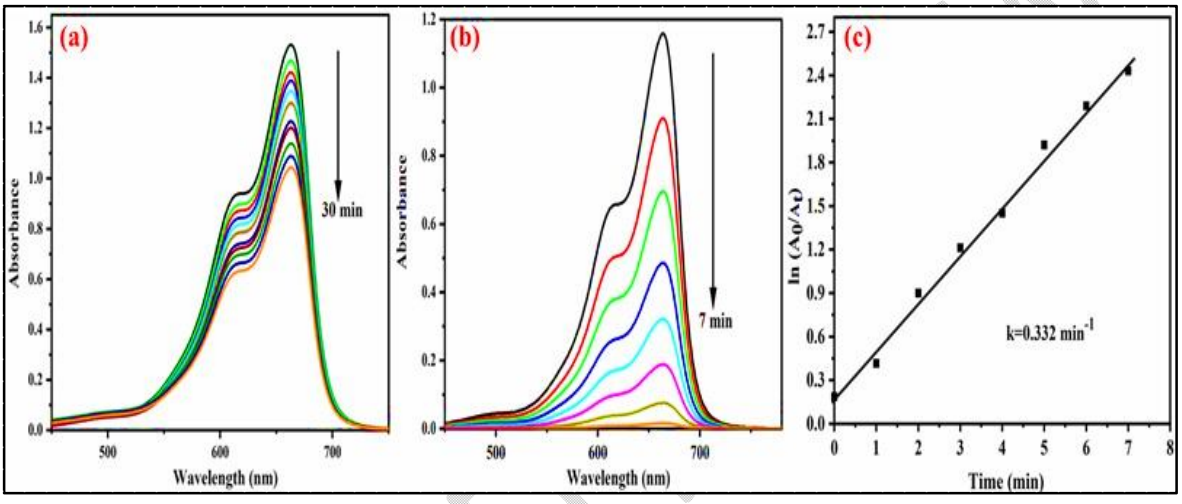

Figure.5 (a) UV-Vis spectrum of reduction of MB by $NaBH_4$ in the absence of CoONPs for a period of 30 min at room temperature. (b) UV-Vis spectrum for the catalytic reduction of MB by $NaBH_4$ in presence of CoONPs. (c) Plot of $\ln(A_0/A_t)$ against time.

3.6. Catalytic reduction of CR

Utilizing UV-Vis spectroscopy, these reduction reactions were tracked. CR exhibits an absorbance peak at 350 nm and 498 nm in aqueous solution[20]. The UV-Vis spectra of CR with $NaBH_4$ in the absence of CoONPs were captured over the course of 30 minutes at room temperature and are shown in **Figure 6a. Figure 6a** depicts a little tendency toward decreasing absorption peak intensity, which suggests a steady reduction in CR[21]. When CoONPs were introduced to the combination of CR and $NaBH_4$, the amount of CR that was absorbing quickly decreased. Within 5minutes, the reduction reaction was finished. The constant decline of the CR absorption peak at 493 nm over time that can be seen in **Figure 6b** indicates that the dye has deteriorated gradually. Furthermore, during the process, there was no peak visible[22]. It was discovered that the decrease of dyes follows pseudo-firs-order kinetics, given that the

concentration of $NaBH_4$ was substantially greater than that of dye (**Figure 6c**), indicating that the reduction of dyes follows pseudo-firs-order kinetics. The rate constant was found to be 0.443 min^{-1}.

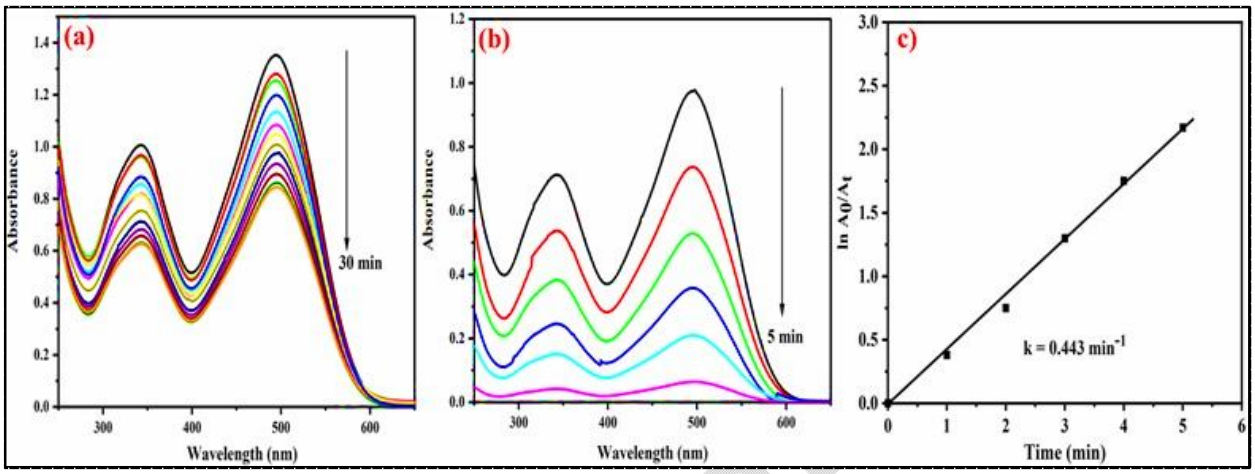

Figure. 6 (a) UV-Vis spectrum of reduction of CR by $NaBH_4$ in the absence of CoONPs for a period of 30 min at room temperature. (b) UV-Vis spectrum for the catalytic reduction of MB by $NaBH_4$ in presence of CoONPs. (c) Plot of $ln(A_0/A_t)$ against time.

3.7. Catalytic reduction of 4-NP

We studied the catalytic reduction of 4-NP using CoONPs as the catalyst and $NaBH_4$ as the reducing agent. 4-NP is well-known for its many beneficial uses, including the manufacture of dyes, pharmaceuticals, photo-graphic developers, agrochemicals, and anticorrosion. The maximum peak of aqueous 4-NP is visible at 317 nm[23]. When $NaBH_4$ was added to 4-NP, the peak was moved to 400 nm, and an intense yellow color was produced as a result of the creation of para nitrophenolate ion. The reaction cannot occur without a catalyst, and the peak persisted for several hours (**Figure7a**). However, the peak at 400 nm declines with the addition of produced CoONPs, and a new peak at 297 nm related to the synthesis of 4-AP appears instead[24]. After 7 min the peak intensity at 400 nm related to 4-NP almost diminished, indicating that the reduction of 4-NP was closely completed. The presence of CoONPs in the reaction supported the transfer of electrons from $NaBH_4$ to the 4-NP nitro group and its reduction to 4-AP. UV-Vis spectroscopy was used to qualitatively monitor this reaction, as illustrated in **Figure 7b**. This research demonstrated the produced CoONPs catalytic activity for 4-NP reduction when distributed in papaya leaf extract. The pseudo first order kinetics equation was

used to determine the rate constant of this catalytic reduction reaction with respect to 4-NP concentration. Because there was a higher initial concentration of $NaBH_4$ than 4-NP, the rate of reduction was independent of $NaBH_4$ concentration[25]. The slope of the plot of $\ln(A_t/A_o)$ over time **(Figure7c)** was used to calculate the rate constant and was found to be 0.330 min^{-1}.

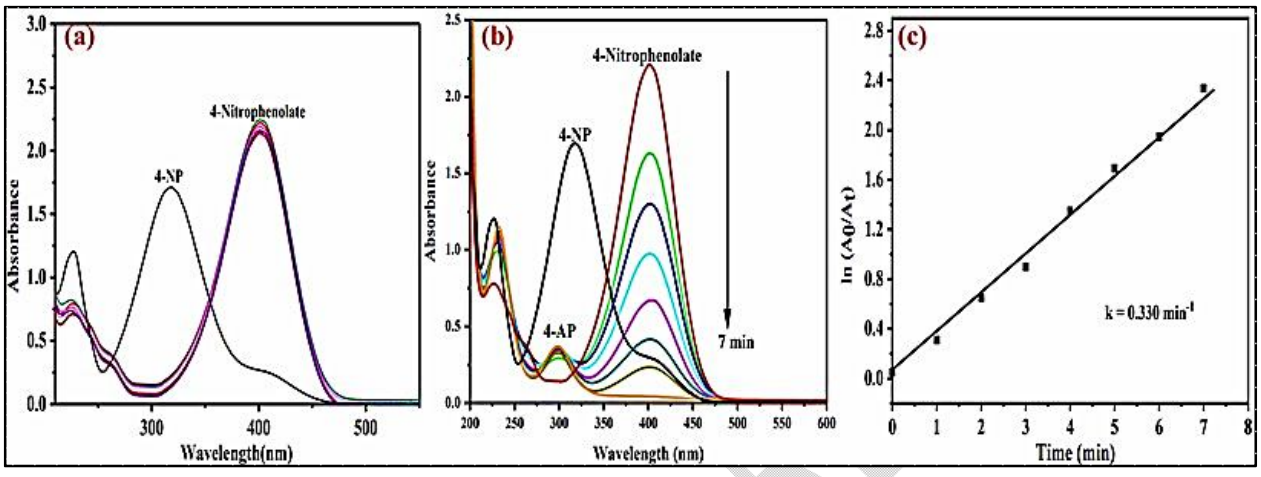

Figure. 7 (a) UV−vis spectra of 4-nitrophenol and 4-nitrophenol with $NaBH_4$ without addition of any catalyst (b)4-nitrophenol with $NaBH_4$ in the presence of CoONPs as catalyst (c) and plot of $\ln(A_0/A_t)$ against the reaction time.

4. CONCLUSIONS

In conclusion, we have effectively established an environmentally friendly microwave irradiation technique for producing CoONPs by reducing $(Co(NO_3)_2.6H_2O$ using papaya leaf extract, a cheap and plentiful natural resource. Papaya leaf extract served as a stabilizing and capping ingredient in this process. The majority of the CoONPs morphology was discovered to be spherical in shape. XRD analysis has verified the creation of extremely crystalline. In the presence of $NaBH_4$, capped CoONPs made from papaya leaf extract were found to be effective as catalysts for the reduction of 4-NP, MB, and CR compounds. It is envisaged that the innovative catalyst will find widespread practical use in the field of organic catalysis. Hence, papaya leaf extract-capped CoONPs are effective catalysts for the treatment of industrial effluents containing toxic contaminants.

5. DISCLAIMER (ARTIFICIAL INTELLIGENCE)

Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc) and text-to-image generators have been used during writing or editing of manuscript.

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