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Synthesis of Cobalt Oxide Nanoparticles and Their Catalytic Application in Esterification Reactions

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A B S T R A C T

Cobalt oxide nanoparticles (CoNPs) have garnered a lot of interest because of their distinctive physicochemical properties and wide catalytic applications. This study reports the synthesis of Co nanoparticles using both green and conventional chemical methods, followed by structural characterization and evaluation of their catalytic performance in esterification reactions. In the green synthesis approach, pomegranate peel extract was employed as a natural reducing and stabilizing agent. The synthesized nanoparticles were characterized using FTIR, SEM, XRD, and UV–Vis spectroscopy. XRD analysis using the Debye Scherrer equation revealed that green-synthesized nanoparticles exhibited smaller crystallite sizes (1–20.4 nm) compared to chemically synthesized nanoparticles (2.6–82 nm). FTIR results confirmed the formation of cobalt oxide through characteristic CoO stretching vibrations. The catalytic activity of the synthesized nanoparticles was evaluated in the esterification of acetic acid with ethanol at 80 °C for 3 hours. The green-synthesized nanoparticles demonstrated superior catalytic performance, achieving 91.32% acid conversion compared to 85.64% for chemically synthesized nanoparticles. While, the effect of reaction time (60, 120, 180 min), at 80 oC on. For the two catalysts used, the acid conversion increased with reaction time. The enhanced catalytic efficiency is attributed to the smaller particle size and the existence of plant-derived biomolecules acting as capping agents. These findings highlight the effectiveness of green synthesis as an eco-friendly and economical efficient method for producing efficient nanocatalysts.

تحضير جسيمات أكسيد الكوبالت النانوية وتطبيقها التحفيزي في تفاعلات الأسترة.

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الـمُخلص

حظيت جسيمات أكسيد الكوبالت النانوية (CoNPs) باهتمام كبير نظرًا لخصائصها الفيزيائية والكيميائية المتميزة وتطبيقاتها الواسعة في مجال التحفيز. تعرض هذه الدراسة تحضير جسيمات الكوبالت النانوية باستخدام كلٍّ من الطريقة الخضراء والطريقة الكيميائية التقليدية، يلي ذلك توصيفها البنيوي وتقييم أدائها التحفيزي في تفاعلات الأسترة.في طريقة التحضير الخضراء، استُخدم مستخلص قشور الرمان كعامل اختزال وتثبيت طبيعي. وتم توصيف الجسيمات النانوية المحضّرة باستخدام تقنيات الأشعة تحت الحمراء بتحويل فورييه(FTIR) ، والمجهر الإلكتروني الماسح(SEM) ، وحيود الأشعة السينية(XRD) ، ومطيافية الأشعة فوق البنفسجية–المرئية.(UV–Vis) أظهرت نتائج تحليلXRD ، باستخدام معادلة ديباي–شيرر، أن الجسيمات النانوية المحضّرة بالطريقة الخضراء تمتلك أحجامًا بلورية أصغر تراوحت بين 1 و20.4 نانومتر، مقارنةً بالجسيمات المحضّرة بالطريقة الكيميائية التي تراوحت أحجامها بين 2.6 و82 نانومتر. كما أكدت نتائج FTIR تكوّن أكسيد الكوبالت من خلال ظهور نطاقات امتصاص مميزة تعود إلى اهتزازات رابطة.Co–O تم تقييم النشاط التحفيزي للجسيمات النانوية المحضّرة في تفاعل أسترة حمض الأسيتيك مع الإيثانول عند درجة حرارة 80 °م ولمدة 3 ساعات. وأظهرت الجسيمات النانوية المحضّرة بالطريقة الخضراء أداءً تحفيزيًا متفوقًا، حيث حققت نسبة تحويل للحمض بلغت 91.32% مقارنةً بـ 85.64% للجسيمات المحضّرة بالطريقة الكيميائية. كما دُرس تأثير زمن التفاعل (60، 120، 180 دقيقة) عند 80 °م، وأظهرت النتائج أن نسبة تحويل الحمض ازدادت بزيادة زمن التفاعل لكلا العاملين الحفازين المستخدمين. ويُعزى التحسن في الكفاءة التحفيزية إلى صغر حجم الجسيمات ووجود الجزيئات الحيوية المشتقة من النبات التي تعمل كعوامل تغليف وتثبيت على سطح الجسيمات النانوية. تُبرز هذه النتائج فعالية التخليق الأخضر بوصفه أسلوبًا صديقًا للبيئة وذا جدوى اقتصادية لإنتاج محفزات نانوية عالية الكفاءة.

الكلمات المفتاحية: الجسيمات النانوية، أكسيد الكوبالت، التخليق الأخضر، التحفيز، الأسترة

1 Introduction

The remarkable physicochemical characteristics of materials at the nanoscale have made nanotechnology one of the most quickly developing areas of contemporary science. Nanoparticles, defined as particles with at least one dimension below 100 nm, demonstrate enhanced surface area, reactivity, and tunable optical and catalytic properties compared to their bulk counterparts. These distinctive characteristics have enabled their application in a broad spectrum of fields including catalysis, medicine, electronics, and environmental cleanup (D. M. Ibrahim, Emrayed, & Youssef, 2025; Nasrllahizadeh, Mahmudi-Goum Yeik, Motaharifar, & Ghaifri Gorab, 2019).

Among different nanomaterials, CoNPs have gained significant attention because of their multiple oxidation states, excellent redox behavior, high stability, and relatively low cost compared to noble metal catalysts (D. M. Ibrahim, Bendaba, & a yaakub Hesien, 2025; Imtiyaz, Singh, & Gaur, 2024). These properties make cobalt oxide nanoparticles promising candidates for heterogeneous catalytic applications, particularly in esterification reactions that serve a critical function in the production of solvents, pharmaceuticals, plasticizers, and fragrances (Bayda, Adeel, Tuccinardi, Cordani, & Rizzolio, 2019).

Traditionally, CoNPs have been prepared using chemical methods such as co-precipitation and thermal decomposition of metal precursors (Baruah, 2023; D. M. Ibrahim, Idris, & Mohammed, 2024). Even though these techniques offer good control over crystallinity and particle size, they typically entail hazardous chemicals and high energy consumption. Recently, Plant extract-based green synthesis techniques have become viable substitutes because of their eco-friendly nature, low cost, and ability to produce stable nanoparticles through naturally occurring reduction and stabilization agents (D. Ibrahim, Abdelghani, Anwagy, & Rizkallah, 2024; Sun et al., 2022).

Bioactive constituents present in pomegranate peel extract, including polyphenols, flavonoids, and other organic compounds, contribute significantly to nanoparticle synthesis by functioning as both reducing and stabilizing agents during the formation process (Khan et al., 2021). Although green synthesis has gained increasing attention, comparative studies evaluating the catalytic execution of green and chemically synthesized CoNPs remain limited.

This research introduces a comparative analysis of CoNPs synthesised using green and chemical approaches, utilising pomegranate peel extract as a natural source of reducing and stabilising compounds. Additionally, this study provides a detailed evaluation of the catalytic performance of both nanoparticles in esterification reactions and analyses the effects of the synthesis approach on structural properties and catalytic efficiency.

2 Materials and Methods

Table 1 illustrates the materials used for the synthesis of cobalt nanoparticles.

Table 1: Materials Used in the Preparation of Cobalt Nanoparticles

Chemicals	Molecular Formula
Methanol	**CH3OH**
Ethanol	**CH3CH2OH**
Plant material	**Pomegranate peels**
Cobalt nitrate hexahydrate	**(Co(NO3)26H2O)**
Sodium hydroxide	**NaOH**
Sodium Chloride	**NaCl**
Hydrochloric acid	**HCl**
Acetic acid (99 %)	**CH3COOH**
Phenolphthalein	**C20H14O4**

2.1 Synthesis of CoNPs

2.1.1 Synthesis of CoNPs via a Green Method

Pomegranate peel extract was applied as a natural reducing and stabilising agent in the green and eco-friendly prepare of CoNPs. All compounds employed in this study were of analytical grade and were applied without additional purification. Throughout the synthesis process, deionized water was utilised. Figure 1 displays the Green Synthesis of CoPNs flowchart.

Pomegranate peels were acquired at a nearby local store, sliced into little pieces, and thoroughly washed with ultrapure water to remove surface pollutants and impurities. An electronic grinder was used to homogenise a known mass of the cleaned peels (20 g) with 150 mL of deionized water. To aid in the extraction of bioactive chemicals, the resultant mixture was heated to around 75 °C while being continuously stirred. The mixture was heated, allowed to cool to ambient temperature, and then filtered to produce a brown aqueous extract that was employed right away to create CoNPs.

To create NPs, a 1 M aqueous solution of cobalt nitrate hexahydrate was mixed with 90 mL of newly made pomegranate peel extract while being continuously stirred. The reaction mixture was heated to around 75 °C until precipitates started to form. To allow for full reaction and particle formation, the temperature was then lowered to 60 °C and the mixture was kept there for

90 minutes. The mixture was then maintained overnight at room temperature to ensure reaction completion.

The precipitates were separated by centrifugation at 14,000 rpm for 10 minutes, then washed three times with ultrapure water and 100% ethanol to eliminate unreacted species and residual contaminants. The washed goods were dried in an oven at 60 degrees Celsius for 8 to 9 hours. After drying, the material was coarsely milled to produce a homogeneous powder, which was then submitted to structural, morphological, and catalytic characterization.

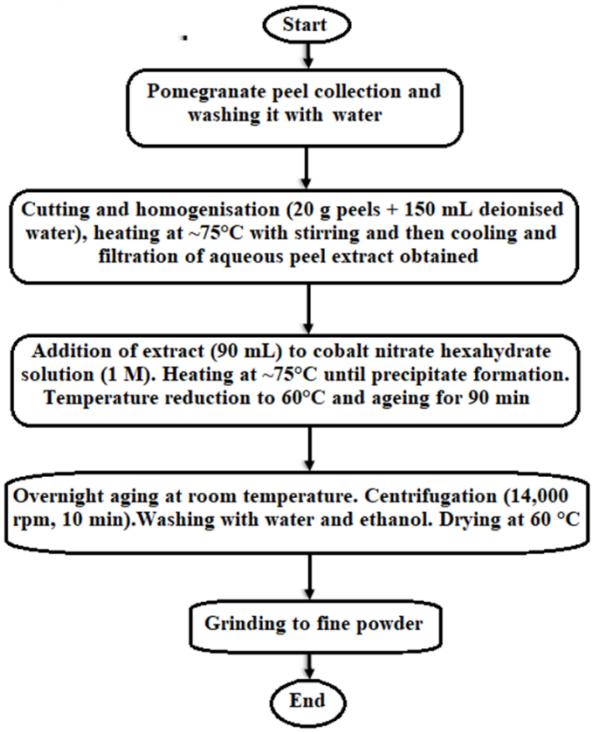


Fig. 1: Flow Chart Synthesis of CoNPs

2.1.2 Synthesis of CoNPs via a Chemical Method

Figure 2 shows the flowchart of the chemical co-precipitation method. Cobalt oxide CoNPs were synthesised using a co-precipitation method without the addition of any capping agents. In this procedure, "cobalt nitrate hexahydrate ($Co(NO_3)_2 \cdot 6H_2O$) and sodium hydroxide (NaOH)" were employed as the precursor and precipitating agent, respectively, at a molar ratio of 1:2. The chemicals were dissolved in 20 mL of distilled water with continuous magnetic stirring to obtain a clear and uniform solution.

Thereafter, a 2 M NaOH solution was gradually introduced dropwise into the cobalt nitrate solution under continuous stirring at ambient temperature. The resulting mixture was stirred for approximately 2 hours to promote the formation of cobalt hydroxide precipitates. Following the reaction, the mixture was left undisturbed overnight to allow complete precipitation. The clear supernatant was then carefully separated by decantation.

The resulting precipitates were separated by centrifugation and then subjected to drying and thermal treatment at 353 K for 5 hours. This thermal treatment led to the formation of black-colored cobalt oxide (CoNPs). The prepared nanoparticles were subsequently collected and used for further structural and morphological characterization.

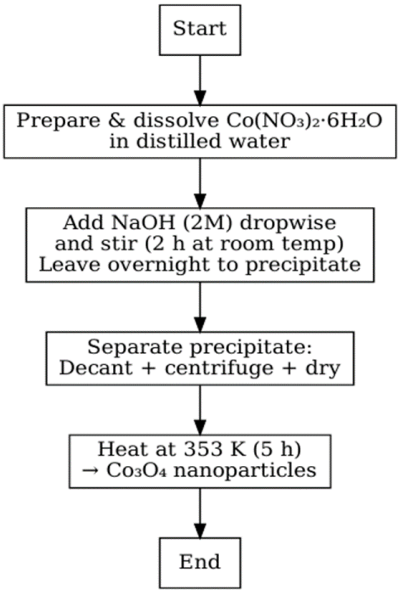


Fig. 2: Flowchart illustrating the chemical co-precipitation method for the prepared of CoNPs

The separation and purification steps involved in both green and chemical synthesis routes were carried out through a filtration process, as illustrated in Figure 3. This step is essential for removing unreacted species and soluble impurities, ensuring the formation of pure CoNPs suitable for further characterization.

Fig. 1: Filtration process of green- and chemically synthesized cobalt oxide NPs

2.2 Characterization Techniques

The synthesised CoNPs were examined by FT-IR, UV-Vis, XRD, and SEM. FT-IR was utilised to find the functional groups contained in the samples. UV-Vis was employed to establish nanoparticle production by their optical behaviour. XRD was applied to investigate the crystalline structure and estimate crystallite size. SEM offered extensive information on surface shape and particle distribution.

2.3 Catalytic Activity

2.3.1 Esterification of Acetic Acid

In a typical reaction, 20 mL of acetic acid and 40 mL of alcohol were introduced into the reaction flask and heated to the desired reaction temperature, which was considered the initial reaction time (t_0). Subsequently, 0.2 g of cobalt oxide nanoparticles was added to initiate the reaction. The esterification procedure was performed for 3 hours under continuous stirring. Samples were withdrawn from the reaction mixture at one-hour intervals and analyzed by titration using standardized 1.0 M NaOH solution to determine the remaining acidity. The conversion of acetic acid was calculated using the following expression:

$$\text{Percentage of conversion } (\%) = X_{acid} = \frac{ai-at}{ai} X\ 100 \quad (1)$$

where (ai) represents the initial acidity of the reaction mixture at time at t0, and (at) denotes the acidity at reaction time at (t).

2.3.2 Conventional Method using One Parameter at Time Approach

This approach was utilized to study the individual effects of key reaction parameters on the esterification process. To investigate the role of the catalyst, esterification reactions were performed either in the absence of a catalyst or in the presence of 0.2 g of cobalt oxide nanoparticles, while maintaining all other reaction conditions constant. The reaction mixture containing acetic acid and alcohol was heated to 80 °C, which was considered the reaction start time. Samples were collected every hour to determine the extent of acetic acid conversion.

The influence of reaction time was evaluated by carrying out esterification reactions for different durations of 60, 120, and 180 minutes under identical reaction conditions. Samples were collected at predetermined intervals to monitor the progression of acetic acid conversion.

3 Result and Discussion

3.1 Physical Properties

Physical properties of the green- and chemically synthesized cobalt nanoparticles are summarized in Table 2. These properties include melting point, color, surface texture, and solubility in various.

Table 2: Comparison of Physical Properties of Green Synthesized and Chemically Synthesized Cobalt Nanoparticles

Nanoparticle	Melting point	Solubility in water	Solubility in alcohol	Solubility in DMS	Solubility in DMF	Colour and texture
Green Co nanoparticles	Over than 350°C	Soluble	Non soluble	Soluble With heating	Soluble With heating	Black hard powder
Chemical Co nanoparticles	Over than 350°C	Soluble	Non soluble	Soluble With heating	Soluble With heating	Pale Red soft powder

3.2 XRD Analysis

Figures 4 and 5 show the XRD patterns of cobalt oxide nanoparticles synthesized employing the green method (pomegranate fruit extract) and the chemical method, respectively. The XRD patterns of both green and chemically synthesized nanoparticles exhibited characteristic diffraction peaks corresponding to the cubic spinel structure of CoNPs, confirming successful nanoparticle formation. The average crystallite size was determined applying the Deibye-Scherer equation.

$$D = \frac{K\lambda}{\beta \cos\theta} \quad (2)$$

Here, D refers to the average size of the crystallites, θ signifies the Bragg angle of diffraction, and λ indicates the wavelength of the applied X-ray radiation. The parameter K represents the dimensionless shape factor, generally assigned a value of 0.94, whereas β corresponds to the FWHM of the relevant diffraction peak expressed in radians.

The average (D) of the green-synthesized CoNPs was estimated to be 20.4 nm at θ = 18.22° and β = 0.43°. In addition, a smaller crystallite size of approximately 1 nm was obtained at higher peak broadening values. For chemically synthesized cobalt oxide nanoparticles, the crystallite size was estimated to be 82 nm at θ = 14.7° and β = 0.111°, while another peak showed a crystallite size of approximately 2.6 nm at β = 3.8°.

The green-synthesized nanoparticles exhibited broader diffraction peaks, indicating smaller crystallite size compared to chemically synthesized nanoparticles. The reduction in crystallite size enhances catalytic performance because increased surface area and higher availability of active catalytic sites. The smaller crystallite size observed in green-synthesized cobalt oxide nanoparticles improves their catalytic efficiency, which correlates well with their enhanced performance in the esterification of acetic acid.

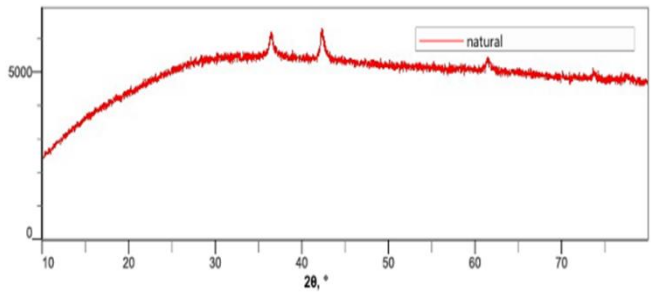


Fig. 4: XRD Pattern of Green-Synthesized Cobalt NPs

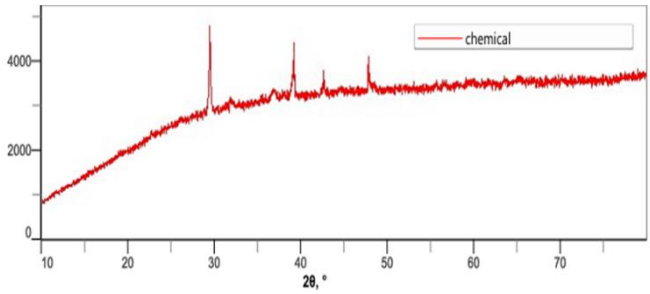
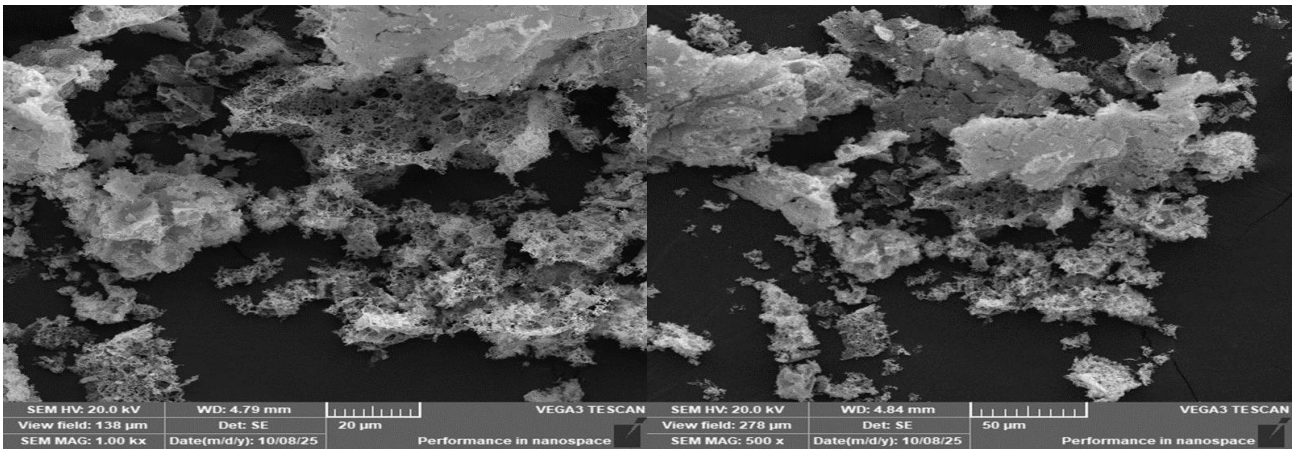


Fig. 5: XRD Pattern of Chemical -Synthesized Cobalt NPs

3.3 SEM Analysis

SEM was used to examine the surface morphology of the synthesized cobalt NPs. Figures 6 and 7 show the SEM images of cobalt NPs synthesized by chemical and green methods, respectively.

In the green synthesis using pomegranate extract, the nanoparticles appeared less distinct in the SEM images due to the presence of bioactive compounds that act as reducing and capping agents on the nanoparticle surface. Despite this, the green-synthesized nanoparticles were smaller in size compared to those synthesized by the chemical method, indicating a higher surface area-to-volume ratio. This feature is advantageous for catalytic applications and highlights the effectiveness of green-synthesized cobalt NPs in the esterification of acetic acid.



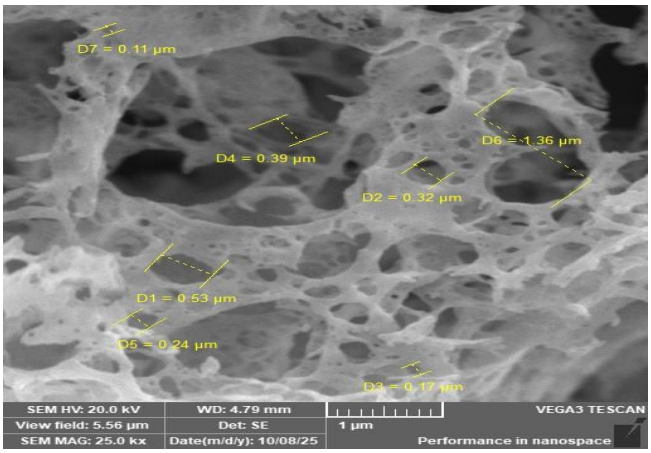
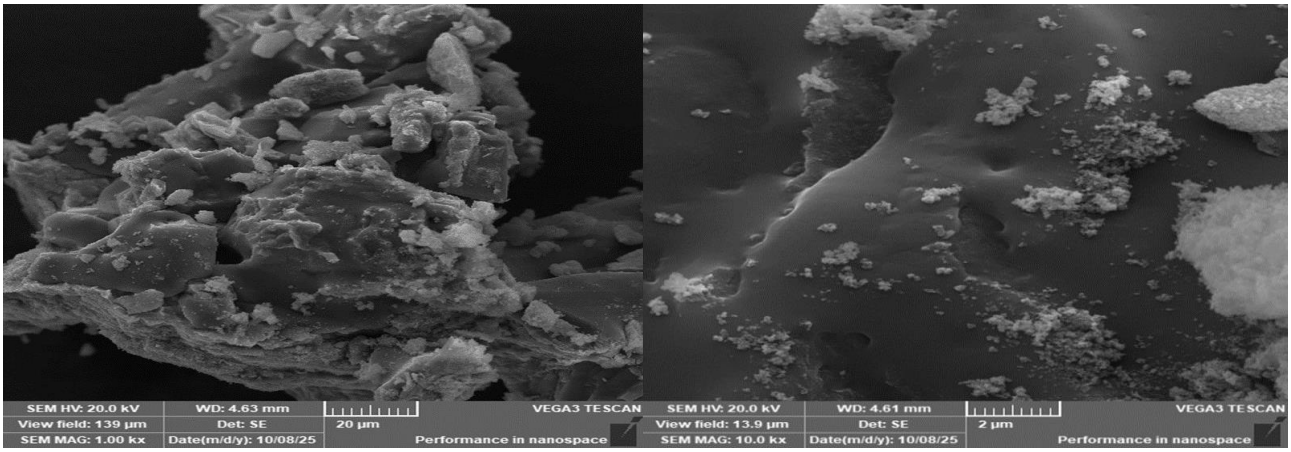


Fig. 6: SEM Image of Chemically Synthesized Cobalt NPs



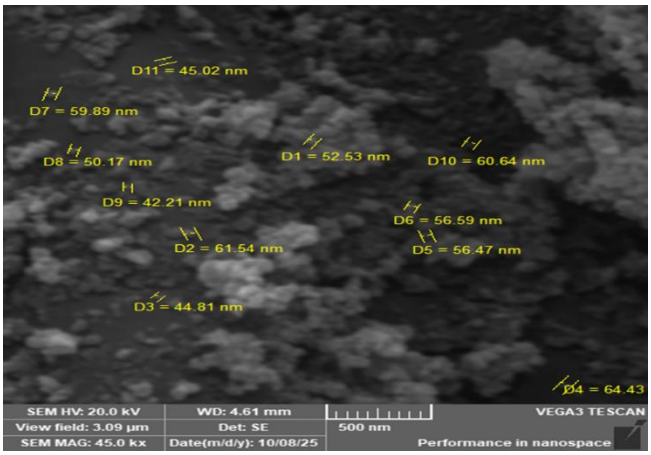


Fig. 7: SEM Image of Green Synthesized Cobalt NPs

3.4 UV–Vis Spectroscopy

The UV–Vis spectra of green- and chemically synthesized cobalt nanoparticles exhibited characteristic absorption bands at approximately 480 nm and 780 nm, with absorbance values of 1.900 and 1.600, respectively.

The absorption band at approximately 780 nm confirms the production of CoNPs. Figures 8 show the UV-Vis spectra of both green and chemically synthesised CoNPs. These findings comply with earlier studies. research that reported the production of CoNPs with distinctive absorption bands in roughly the same wavelength range (Yugandhar, Vasavi, Uma Maheswari Devi, & Savithramma, 2017).

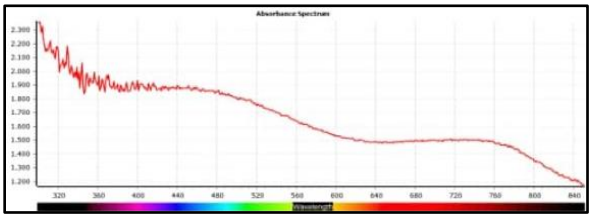


Fig. 8: UV-visible spectrum analysis

3.5 Infrared

Figure 9 shows the FT-IR spectrum of the cobalt oxide sample prepared by the chemical method. Abroad peak appears at 3617.61 cm, which is attributed to the bond stretching vibration O-H. The presence of absorption bands at 423.72-572.72 cm indicates different modes of bands of bond bending vibration Co-O. The peak at 1525.32 cm indicates the Co-O band. Stretch vibration of the complete cobalt nanoparticles.

When comparing these results with previous studies, it was noted that these spectroscopic results for Nano sized cobalt oxide samples are close to the FTIR spectroscopic results for the Nano sized copper oxide sample synthesised by the chemical method (Raul et al., 2014).

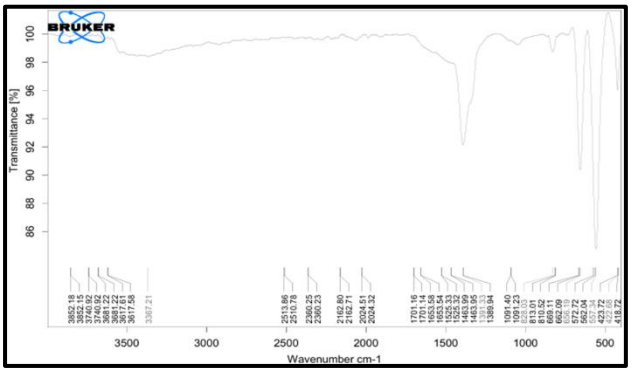


Fig. 9: Absorption spectrum of Chemical Cobalt CoNPs

Figure 10 presents the FTIR spectrum of cobalt oxide synthesized via the green method. The bands at 3617.77, 2507.95, 1462.23, and 1090.56 cm^{-1} are assigned to O–H, C=O, C=C, and C–O stretching vibrations, respectively. The absorption at 2507.95 cm^{-1} is also related to C≡C stretching, while the band at 1090.56 cm^{-1} corresponds to symmetric and asymmetric C–OH bending vibrations and C–H stretching of methyl groups. The peak at 1525.32 cm^{-1} is attributed to C–N stretching vibrations of amine groups. Additionally, the bands at 810.52 and 822.21 cm^{-1} are associated with C–C vibrations, whereas the peaks in the range of 426.34–564.28 cm^{-1} correspond to Co–O stretching vibrations. The detected functional groups confirm the presence of polyphenolic compounds from pomegranate extract, which are most likely used as reducing and stabilising agents during nanoparticle formation.

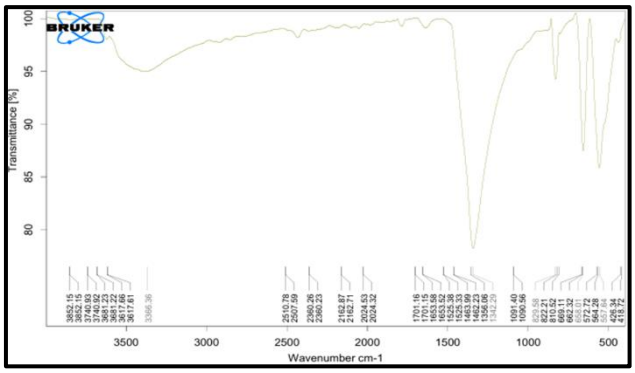


Fig. 10: Absorption spectrum of Green Cobalt CoNPs

3.6 Optimization of Esters Produced via the Conventional Method

3.6.1 Effects of catalysts

The catalytic activity of cobalt oxide NPs was investigated using esterification reaction of acetic acid and ethanol. Figure 11 shows the esterification of acetic acid and ethanol with two catalysts (green and chemical synthesised cobalt oxide) at 80 oC for 3 hours. The green synthesised cobalt oxide produced a high acid conversion of 91.32%, in contrast, the acid conversion using chemical synthesised cobalt oxide was 85.64%.

This indicates that the green synthesised cobalt oxide exhibited higher catalytic activity compared with chemical synthesised cobalt oxide, this enhanced activity is attributed to the presence of plant biomolecules as surface capping agent, which increased the catalytic active site and facilitate the esterification reaction. From the SEM analysis of green synthesised cobalt oxide indicate of the presence of this bioactive compound on the surface of cobalt oxide NPs surface.

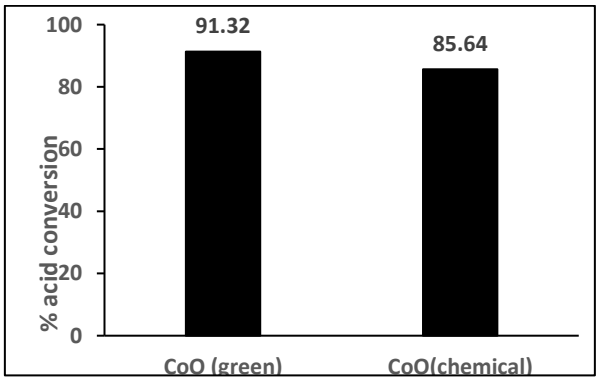


Fig. 11: Effect of catalyst on esterification of acetic acid with methanol at 80 oC after 3 hours

3.6.2 Effects of reaction time

Figure 12, illustrates the effect of reaction time (60, 120, 180 min), at 80 oC on acetic acid conversion catalysed by green and chemical synthesized CoO. For the two catalysts used, the acid conversion increased with reaction time. Green synthesized CoO exhibited higher conversion at all the reaction times, reaching 91.32 %

after 180 min compared to 85,64 % for chemically synthesized. This was attributed to the surface properties and increased the active sites. In general, the figure shows the relationship between the reaction time and acid conversion percentage using the green synthesized cobalt oxide, where the increase in the reaction time leads to gradual increase in acid conversion. For the esterification reactions; a gradual increase in acid conversion is expected as the reaction time increase (Almadani, Radzi, & Harun, 2016). Also the esterification reaction is a reversible reaction, thus the 180 minutes chosen to be the optimum reaction time (Syazwani, Rashid, Mastuli, & Taufiq-Yap, 2019).

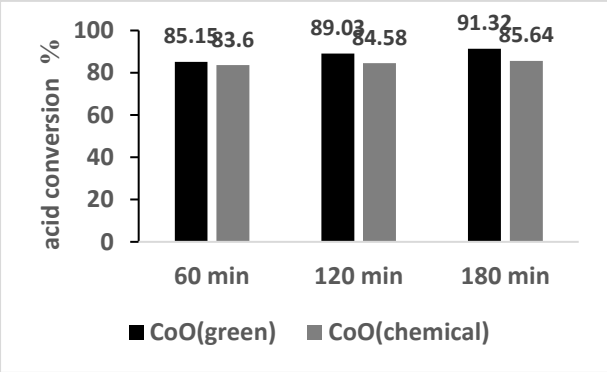


Fig. 12: Variation in Esterification Efficiency of Acetic Acid and Methanol as a Function of Reaction Time at 80 °C

The obtained acid conversion efficiency (91.32%) using green-synthesized CoO nanoparticles is comparable or superior to previously reported cobalt-based catalysts used in esterification reactions. Several studies have reported conversion efficiencies ranging between 70-88% depending on catalyst preparation method and reaction conditions, indicating that the present green synthesis route provides enhanced catalytic activity (Andualem, 2020; Baruah, 2023; Kumar et al., 2025).

4 Conclusions

This study demonstrated that the synthesis method plays a significant role in determining the structural and catalytic properties of cobalt oxide nanoparticles. The green synthesis approach using pomegranate peel extract successfully produced CoO nanoparticles with smaller crystallite size and improved catalytic efficiency compared to conventional chemical synthesis. The enhanced catalytic performance is attributed to increased surface area and the stabilizing effect of plant-derived biomolecules. The results showed the potential of green synthesis as a sustainable strategy for developing efficient nanocatalysts for industrial esterification processes.

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