ISETC2025

2 ND INTERNATIONAL SCIENCE, ENGINEERING AND TECHNOLOGY COLLOQUIUM

THE POWER OF SCIENTIFIC INNOVATION

INTELLIGENT SOLUTIONS FOR A SUSTAINABLE WORLD

ORGANIZED BY:
FACULTY OF APPLIED SCIENCES
UNIVERSITI TEKNOLOGI MARA
PERAK BRANCH, TAPAH CAMPUS
MALAYSIA

CO-ORGANIZED BY:
三重大学
MIE UNIVERSITY

2nd International Science, Engineering and Technology Colloquium
(ISETC 2025)
Faculty of Applied Sciences,
Universiti Teknologi MARA,
Perak Branch, Tapah Campus,
35400 Tapah Road, Perak, Malaysia.

Colloquium date: 9th July 2025
Publication date: 9th September 2025

Proceedings of Extended Abstracts

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e-ISBN: 978-629-97630-5-5

Published by Faculty of Applied Sciences, Universiti Teknologi MARA,
Perak Branch, Tapah Campus, 35400 Tapah Road, Perak, Malaysia.

PREFACE

Welcome to the 2nd International Science, Engineering and Technology Colloquium 2025 (ISETC 2025)!

It is with great pleasure that we present this year's colloquium, a distinguished gathering of scholars, researchers, and professionals dedicated to the advancement of applied sciences. Held virtually on 9th July 2025, ISETC 2025 serves as a platform to highlight cutting-edge research, foster meaningful networking, encourage collaborations, and explore innovative solutions to global challenges.

This year's theme, "The Power of Scientific Innovation: Intelligent Solutions for a Sustainable World" highlights our shared goal to solve global challenges through applied science. It celebrates innovation as a key to building a more sustainable and resilient future. We're proud to feature keynote speakers and presenters who bring fresh insights, bold ideas, and breakthroughs shaping tomorrow's world.

We would like to extend our heartfelt appreciation to the Faculty of Applied Sciences, Universiti Teknologi MARA, Perak Branch, Tapah Campus, for their outstanding support and assistance throughout the planning and execution of the event. Their dedication has been instrumental to the success of this colloquium. We would like to sincerely thank all participants and researchers for their valuable contributions. We also appreciate the reviewers for their careful evaluations and helpful feedback. Your hard work and support mean a lot to us.

As editors, we are proud to present this collection of proceedings. It is an honour to bring together the interesting and meaningful work of our speakers and authors. We hope the ideas shared will lead to new discoveries, encourage more research, and help solve global challenges through science and innovation.

Warm regards,

Dr. Emi Norzehan Mohamad Mahbob
Dr. Saiyidah Nafisah Hashim
Ms. Lili Widarti Zainuddin
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ISETC 2025 Proceedings Editors

EDX ANALYSIS OF GOLD NANOPARTICLES SYNTHESIZED BY TURKEVICH METHOD AT 100°C WITH 2.8 MOLAR RATIO

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Abstract: This study presents the elemental characterization of gold nanoparticles (AuNPs) synthesized using the Turkevich method under optimized conditions. The Turkevich method, involving the reduction of chloroauric acid ($HAuCl_4$) by sodium citrate, is widely employed for AuNP synthesis due to its simplicity and reproducibility. Gold nanoparticles were synthesized at 100°C using a molar ratio of 2.8 (citrate to gold precursor) and subsequently characterized using Energy Dispersive X-ray Spectroscopy (EDX) to determine their elemental composition and purity. The objective of this research was to analyze the elemental composition of the synthesized AuNPs and evaluate the effectiveness of the synthesis conditions in producing nanoparticles of satisfactory elemental purity. EDX analysis was performed on samples prepared on glass slides to minimize substrate interference. Results demonstrated that the synthesized AuNPs contained 81.50 wt% gold as the primary constituent, with carbon (14.31 wt%) and oxygen (3.24 wt%) representing organic residues from the citrate capping agent. Sodium content was minimal at 0.95 wt%, while chloride was completely absent (0.00 wt%), indicating complete reduction of the gold precursor. The satisfactory gold content and absence of chloride contamination confirm the effectiveness of the synthesis conditions employed. The presence of carbon and oxygen is attributed to the citrate stabilizing layer, which is essential for nanoparticle stability and prevents aggregation. These findings validate the Turkevich method as an effective approach for producing gold nanoparticles with adequate elemental purity suitable for various applications.

Keywords: *Gold nanoparticles, Turkevich method, EDX analysis, elemental composition, nanoparticle characterization*

INTRODUCTION

Gold nanoparticles (AuNPs) have attracted considerable attention in nanotechnology due to their unique physicochemical properties, including surface plasmon resonance, high surface area-to-volume ratio, and excellent biocompatibility. These properties make AuNPs valuable for diverse applications in biomedicine, catalysis, and sensing technologies (Bai et al., 2020; Kapil et al., 2021; Milan et al., 2022). The synthesis of high-quality AuNPs with controlled size, shape, and composition is crucial for their successful application in these fields. The Turkevich method, first developed in 1951 and later refined by Frens, remains one of the most widely used chemical reduction approaches for AuNP synthesis. This method involves the reduction of tetrachloroauric acid ($HAuCl_4$) by sodium citrate in aqueous solution, where citrate functions as both a reducing agent and a stabilizing agent. The citrate ions not only facilitate the reduction of Au^{3+} to Au^0 but also provide electrostatic stabilization to prevent particle aggregation through the formation of a protective layer around the nanoparticles. The success of the Turkevich method depends on several parameters, including temperature (Bari et al., 2023; Gul et al., 2022), pH (Yazdani et al., 2021), and molar ratio of reactants (Bahmanyar et al., 2023; Dong et al., 2020). Optimal synthesis conditions are essential to achieve complete reduction of the gold precursor and minimize contamination from unreacted starting materials. Characterization of the elemental composition provides crucial information about the purity and quality of the synthesized nanoparticles. This study aimed to: (i) synthesize AuNPs using the Turkevich method under optimized conditions (100°C, 2.8 molar ratio), (ii) characterize the elemental composition of the synthesized AuNPs using EDX spectroscopy, and (iii) evaluate the purity and quality of the resulting nanoparticles based on their elemental analysis. Understanding the elemental composition is fundamental for assessing the suitability of synthesized AuNPs for specific applications and ensuring their safety and effectiveness.

MATERIALS AND METHODS

Materials

Gold(III) chloride solution (99.99% trace metals basis, 30 wt. % in dilute HCl) and Trisodium citrate dihydrate ($C_6H_9Na_3O_9$) were obtained from commercial suppliers. Deionized (DI) water was used throughout the synthesis process. All chemicals were used without further purification.

Synthesis Procedure

Gold nanoparticles were synthesized using the Turkevich method under optimized conditions. A 0.25 mM solution of $HAuCl_4$ (50 mL) was prepared in DI water and heated to 100°C under constant magnetic stirring at 1000 rpm. Upon reaching the target temperature, sodium citrate solution was rapidly added to achieve a molar ratio of 2.8 (citrate to gold precursor). The reaction mixture was maintained at 100 °C with continuous stirring until a distinct color change from pale yellow to ruby red was observed, indicating the successful formation of gold nanoparticles.

Sample Preparation

After synthesis, the AuNP solution was allowed to cool at room temperature naturally before being stored at 4 °C until further use. For EDX analysis, a 50 µL volume of the stored AuNP solution was drop-cast onto a clean glass slide and left to dry at ambient conditions for 48 hours. The dried slide was then baked at 100 °C for 15 minutes to enhance adhesion to the surface. The prepared samples were subsequently mounted for Ultra-High Resolution Scanning Electron Microscopy (UHR-SEM) and EDX analysis to determine the elemental composition of the synthesized nanoparticles. No accurate particle sizing was performed, as the focus of this study was limited to assessing elemental composition rather than detailed morphological characterization.

EDX Characterization

EDX analysis was performed using an UHR-SEM equipped with an Ultim Extreme EDX detector. The analysis was conducted at an accelerating voltage of 3.00 kV to minimize beam-induced damage while ensuring sufficient excitation of characteristic X-rays. The working distance was maintained at 3.9 mm, with a magnification of 25,000×. Spectrum 13 was selected as representative of the typical elemental composition of the synthesized AuNPs. Quantitative elemental analysis was performed on this spectrum to determine the weight percentages of all detected elements.

RESULTS AND DISCUSSION

The EDX analysis of the synthesized gold nanoparticles revealed the elemental composition presented in Table 1, which demonstrates the successful synthesis of AuNPs with satisfactory gold content.

Table 1. Elemental Composition of Gold Nanoparticles Synthesized at 100°C with 2.8 Molar Ratio

Element	Weight Percentage (wt%)
Carbon (C)	14.31
Oxygen (O)	3.24
Sodium (Na)	0.95
Chloride (Cl)	0.00
Gold (Au)	81.50

The results demonstrate that gold constitutes the major component of the synthesized nanoparticles at 81.50 wt%, confirming the successful reduction of the gold precursor and formation of metallic gold nanoparticles. This acceptable gold content indicates effective synthesis conditions and good conversion efficiency from the gold salt precursor to metallic nanoparticles. The complete absence of chloride (0.00 wt%) is particularly significant as it indicates complete reduction of the $HAuCl_4$ precursor. The presence of residual chloride would suggest incomplete reduction and potential contamination from unreacted starting materials. The zero-chloride content validates the effectiveness of the synthesis temperature (100°C) and molar ratio (2.8) in achieving complete precursor conversion. The presence of carbon (14.31 wt%) and oxygen (3.24 wt%) is attributed to the citrate molecules that serve as both reducing and capping agents. These organic components form a protective layer around the gold nanoparticles. The negatively charged carboxylate groups of citrates are responsible for the charge stabilization of the AuNPs (Grys et al., 2020), which helps prevent aggregation and ensure colloidal stability. Sodium content was minimal at 0.95 wt%, representing residual sodium from the citrate reducing agent. Despite the absence of a post-synthesis purification step, the low sodium content suggests limited incorporation of excess reactants into the final product. The satisfactory gold purity combined with the absence of chloride contamination demonstrates that the chosen synthesis conditions (100°C, 2.8 molar ratio) are effective for producing gold nanoparticles of good quality. Although there is potential to improve gold purity toward 90%, the current results demonstrate successful synthesis suitable for further applications.

Figure 1 presents the scanning electron microscopy image of the synthesized gold nanoparticles deposited on a glass slide substrate. The SEM image reveals a heterogeneous distribution of nanoparticles across the surface, with particles appearing as bright white spots against the darker background of the glass substrate. The nanoparticles appear to exhibit a generally spherical to near-spherical morphology, which is commonly associated with AuNPs synthesized via the Turkevich method. However, due to insufficient magnification, the shape could not be precisely determined. The particle size is presumed to be in the nanometer range, consistent with typical Turkevich-synthesized gold nanoparticles; however, accurate estimation was not performed, as the primary focus of this study was on assessing the elemental composition rather than detailed morphological characterization. The image shows both individual nanoparticles and some degree of particle clustering or aggregation, which often results from the sample preparation process when nanoparticle suspensions are dried on substrates for electron microscopy analysis. This clustering can be attributed to surface dewetting and the coffee-ring effect that occurs during the drying process, leading to the formation of aggregates that may not represent the true dispersion state of the original suspension (Michen et al., 2015). Despite this clustering, individual particles remain distinguishable, indicating that the citrate capping agent provides sufficient stabilization to prevent the nanoparticles from merging into larger, irregular structures. A more detailed morphological characterization, however, would be necessary in a separate study to confirm these preliminary observations. The EDX analysis was performed at the location marked as "Spectrum 13" in Figure 1, which represents a cluster of nanoparticles. This analysis point was strategically chosen to ensure representative sampling of the synthesized material while avoiding areas of the bare glass substrate that could introduce substrate-related artifacts in the elemental analysis.

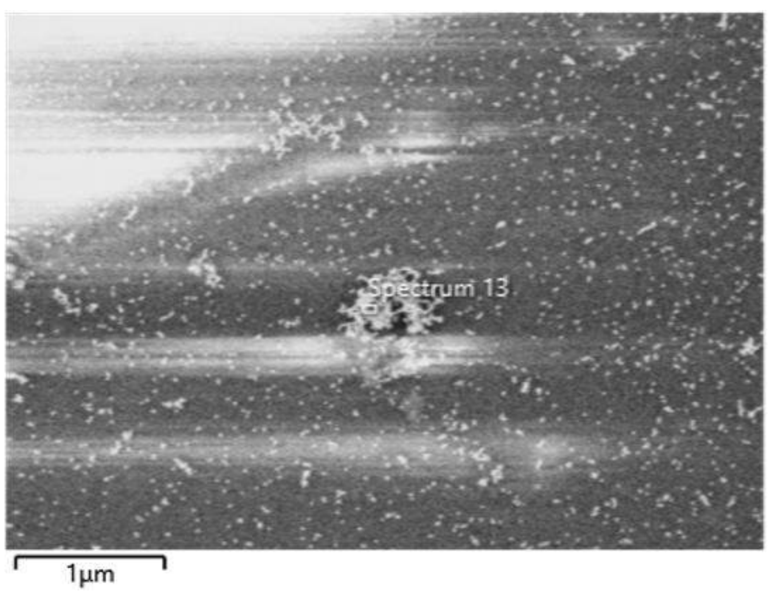

Figure 1. Electron Microscopy Image of Synthesized Gold Nanoparticles with EDX Analysis Point (Spectrum 13)

CONCLUSIONS

This study successfully demonstrated the synthesis and characterization of gold nanoparticles using the Turkevich method under optimized conditions. EDX analysis revealed that the synthesized AuNPs contained 81.50 wt% gold, confirming the notable elemental purity of the nanoparticles. The complete absence of chloride (0.00 wt%) indicates complete reduction of the gold precursor, validating the effectiveness of the synthesis conditions employed. The presence of carbon (14.31 wt%) and oxygen (3.24 wt%) corresponds to the citrate capping layer, which plays a critical role in stabilizing the nanoparticles and preventing aggregation. Although no post-synthesis purification was performed, the low sodium content (0.95 wt%) suggests limited incorporation of excess reactants into the final product. While the current synthesis conditions (100 °C, 2.8 molar ratio) produced gold nanoparticles with acceptable elemental purity, the gold content reached only 81.50 wt%. Future work should explore variations in synthesis parameters such as temperature, reaction time, and citrate-to-gold ratios to further increase gold purity, ideally approaching 90 wt%, to enhance the suitability of the nanoparticles for high-purity applications. Further studies should also include detailed investigation of particle size distribution and shape. High-resolution transmission electron microscopy (HR-TEM) would be appropriate for accurately characterizing particle size and morphology. Advanced surface analysis techniques such as X-ray photoelectron spectroscopy (XPS) could offer valuable information on surface chemistry and oxidation states of elements. Long-term stability studies would also be beneficial for evaluating the durability of the citrate capping layer and its impact on nanoparticle behaviour over time.

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Sekian, terima kasih.

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Saya yang menjalankan amanah,

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Timbalan Ketua Pustakawan

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Setuju.

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