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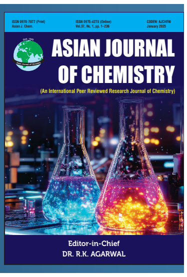

Catalytic Application of Benedict Reaction Waste in Laboratory Wastewater Treatment and Recycling: A Circular Chemical Economy

Randhir Rai*, Rangineni Vennela, Mohith Thalari Adarath and Bhuvanesh Kumar Himachalam

School of Interwoven Arts and Science (SIAS), Krea University, Sri City-517646, India

*Corresponding author: E-mail: randhir.rai@krea.edu.in

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In this study, cuprous oxide was isolated from the Benedict reaction waste and characterized by powder X-ray diffraction and scanning electron microscopic techniques. The isolated material was used as a catalyst for the reductive decomposition of methyl orange using sodium borohydride as a reducing agent. *In situ* formed copper from cuprous oxide catalyzed the dye decomposition reaction and its catalytic performance was compared with commercial copper powder. The catalyst was recycled and reused up to five catalytic cycles without losing its catalytic activity. The same material was also used as a catalyst for treating laboratory wastewater contaminated with methyl orange to promote a circular economy and sustainable waste management in academia. The unused Cu_2O was further converted to Benedict's reagent to increase the net life cycle of copper.

Keywords: Catalysis, Circular economy, Waste management, Wastewater treatment.

INTRODUCTION

Water is one of the basic needs without which life cannot exist. As the population increases, the linear approach to resource consumption has led to water contamination and has impacted all life forms on earth [1,2]. Wastewater contaminated with heavy metals [3], dyes [4] and pharmaceuticals [5] is regarded as extremely hazardous to the environment. To reduce the toxicity of wastewater on the environment, its treatment is mandatory to ensure the treated water is healthy enough to be discharged into the environment.

To control environmental pollution and promote sustainable development, experts developed the concept of a circular economy and the 3Rs (reduce, reuse and recycle) principle [6]. Within the circle of circular economy, copper has been given special attention as it has been known to serve mankind since ancient times and in the present era of advanced technology, its scarcity is expected to begin in the years 2039-2054 [7]. Therefore, the process of recycling, reusing, and upcycling copper from various waste sources can contribute to extending the overall life cycle of copper. Increasing the net life cycle of copper will lead to pollution control and resource conservation. Many researchers have synthesized metal-based catalysts from various waste sources and used them for wastewater treatments [8-10]. Several studies reveal the synthesis of copper-based nanoparticles and their utilization in the treatment of wastewater. Nascimento *et al.* [11] synthesized Cu-Cu_2ONPs from electronic wastes (e-wastes) and used it as a catalyst for decomposing textile dyes in wastewater. Similarly, Wang *et al.* [12] synthesized Cu-coated carbon and studied its Fenton-like catalytic potential towards the decomposition of rhodamine B dye. CuNPs synthesis from e-waste and its application in surfactant contaminated wastewater treatments was also reported by Martins *et al.* [13]. Copper recovery from the industrial waste is also well illustrated in the literature [14]. Copper recovery from the academic laboratories and its application for wastewater treatment are yet to be explored and have a broad future scope towards sustainable laboratory waste management.

Large quantities of copper-based waste are significantly generated in academic laboratories during the educational process. Copper-based reagents such as Fehling's reagents [15], Barfoed's reagents [16] and Benedict's reagents [17] are commonly used reagents for qualitative and quantitative estimation of reducing sugars. When reacting with reducing sugar, Cu(II) gets reduced to Cu(I) and precipitates a Cu_2O, which is generally discarded as waste. Exploring the diverse catalytic capabilities of copper-based materials, the click catalytic potential of Cu_2O isolated from Fehling's reaction waste has already been documented [18,19].

Thus, in continuation of our interest in this field, Cu_2O was isolated from Benedict's reaction waste and used as a catalyst for reductive decomposition of methyl orange and methyl orange contaminated laboratory wastewater treatment. The unused Cu_2O was converted to Benedict's reagents to promote a circular economy practice in academic laboratories and sustainable chemical education processes.

EXPERIMENTAL

Benedict's reagent, methyl orange, sodium borohydride, sodium carbonate and copper powder were purchased from Loba Chemie, India, whereas sodium citrate was purchased from Sigma-Aldrich, USA. Cu_2O was extracted from the waste generated during the practical laboratory class conducted by the undergraduate students in the Benedict's reaction. Distilled water was used as a solvent during the dye decomposition test experiments. Methyl orange-contaminated wastewater was also generated by the students during the practical courses while performing acid-base titration experiments.

Characterization: Isolated Cu_2O and *in situ* formed Cu-particles during dye decomposition reaction were characterized by powder X-ray diffraction (PXRD) technique, using Bruker D8 Advance instruments with Cu$K\alpha$ (λ = 1.5406 Å) radiation in the reflection mode. The morphology and size of the Cu_2O and Cu-powder were analyzed by scanning electron microscope (SEM) using FEI Quanta FEG 200-High resolution scanning electron microscope. Dye decomposition reaction and wastewater treatment were monitored by UV-visible spectroscopic technique using a UV-3200 spectrophotometer.

Extraction of Cu_2O from Benedict's reaction waste: The waste was generated during the practical sessions where undergraduate students qualitatively estimated reducing sugar. Yellow to orange colour Cu_2O formation occurs when Benedict's reagent is heated in the presence of reducing sugar. With the cooperation of students, the waste was collected in a separate beaker and after class, it was centrifuged at 3000 rpm for 30 min. The supernatant was discarded and the Cu_2O was redispersed in 10 mL water and again separated by centrifugation at 3000 rpm for 30 min. The washing was repeated three times and Cu_2O was dried at 50 ºC for 24 h.

Procedure for methyl orange decomposition: A stock solution of methyl orange was prepared by dissolving 50 mg of methyl orange in 1 L of water. A methyl orange solution (100 mL) was taken in a 100 mL beaker containing 5 mg Cu_2O and 10 mg of $NaBH_4$ was added and then magnetically stirred at room temperature. At a time interval of 5 min, 0.3 mL of the reaction mixture was transferred to a quartz cuvette containing 2.7 mL of water and mixed and absorption spectra were recorded. The solution became completely colourless in 30 min. The same reaction was performed using commercial copper powder as a catalyst and the complete decomposition of the dye took place in 30 min. The blank experiments were performed without any catalyst and by keeping all other reaction parameters fixed, no significant decomposition of dyes was observed.

Recyclability of catalyst: To examine the recyclability of catalyst, we performed the methyl orange decomposition reaction using Cu_2O and then absorbance was recorded at the beginning (0 min) and after 30 min. At the beginning and end of the reaction, 0.3 mL of the reaction mixture was taken in a quartz cuvette containing 2.7 mL of water and mixed and the absorbance at 466 nm was measured. After the first cycle, the reaction mixture was kept aside without stirring for 2.0-2.5 h. Once the *in situ* formed Cu powder settled at the bottom of beaker, the water was slowly decanted and 100 mL of methyl orange solution was added and kept on a magnetic stirrer. Then, 10 mg of $NaBH_4$ was added and stirred for 30 min and absorbance was measured at 466 nm at time zero and after 30 min. The catalyst was recycled and reused up to five catalytic cycles without significantly losing its catalytic potential.

Wastewater treatment: Methyl orange-contaminated wastewater was generated during the practical sessions, where the students performed acid-base titration using methyl orange as an indicator. The red coloured wastewater (1.5 L, pH 3) was transferred to a 2 L crystallizing dish, which was neutralized (pH 7) by adding 300 mg of sodium carbonate with continuous stirring and its colour changed to yellow. Then, 50 mg of Cu_2O was added, followed by 100 mg of $NaBH_4$ with constant stirring. Within a minute, a decoloration of the wastewater was observed. The completion of the reaction was confirmed by measuring the UV-visible spectra of the reaction mixture and no absorption corresponding to methyl orange was observed.

Preparation of Benedict's reagent from waste-Cu_2O: A 0.5 g of Cu_2O, 10 mL distilled water and 1 mL of 35 % aqueous H_2O_2 was mixed with continuous stirring. Then, 0.5 mL of conc. H_2SO_4 was added dropwise with constant stirring and within 5 min, a blue colour solution of $CuSO_4$ was obtained. In another 100 mL beaker, 13 g of sodium citrate, 8 g of sodium carbonate and 90 mL of distilled water were mixed. The mixture was stirred till sodium citrate and sodium carbonate dissolved completely. The alkaline sodium citrate solution was added to the copper sulphate solution and a deep blue solution of copper citrate was obtained (Benedict's reagent).

RESULTS AND DISCUSSION

Isolation of Cu_2O from Benedict reaction waste: The Benedict reagent is an alkaline solution of Cu(II)-citrate complex (a complex mixture of copper sulphate, sodium citrate and sodium carbonate) and used as a reagent for a qualitative test to confirm whether the test sugar sample is reducing or non-reducing sugar [17]. In the test experiment, a small quantity of sugar sample was added to the Benedict solution and dissolved. After heating the mixture for a few minutes in the hot water bath, a yellow to orange colour Cu_2O precipitation occurs due to the redox reaction between alkaline Cu(II) and reducing sugar. Generally, the formed Cu_2O was discarded as waste. The same waste generated by the students during their practical classes was collected in a separate container and later isolated by centrifugation (Fig. 1). The isolated waste-Cu_2O was characterized through PXRD, and its shape and morphology were analyzed using SEM (Fig. 2). In the PXRD spectrum (Fig. 2a), the 2θ corresponding to Cu_2O was observed and the obtained

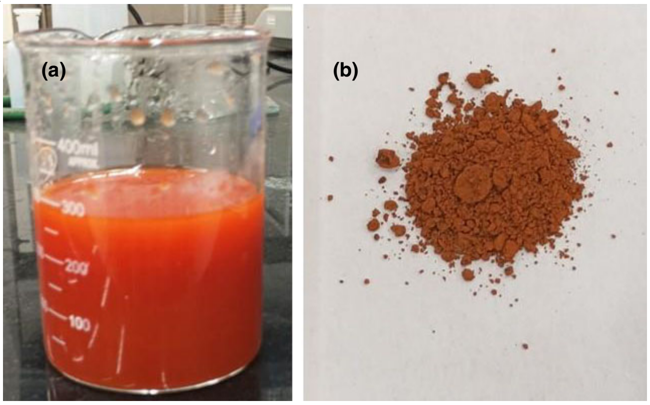


Fig. 1. (a) Benedict's reaction waste and (b) Cu_2O recovered from Benedict's reaction waste

data are consistent with the reported data [18,19]. In SEM image, the particle size appears to be 4-5 μm (Fig. 2b).

Catalytic application of *in situ* formed Cu-powder from waste Cu_2O: Several reports use copper-based materials as catalysts for the decomposition of methyl orange in water. In almost all cases, resources are consumed to synthesize efficient catalysts [20-22]. Considering the sustainability and circular economy viewpoint to evaluate the possible use of waste-Cu_2O is essential for resource conservation. Hence, we evaluated the possible catalytic application of waste-Cu_2O towards the reductive decomposition of methyl orange.

Methyl orange solution containing Cu_2O was treated with $NaBH_4$ with continuous stirring at room temperature. The Cu_2O gets reduced to Cu and *in situ* formed Cu catalyzes the reductive decomposition reaction of methyl orange. The Cu_2O (5 mg) and $NaBH_4$ (10 mg) took approximately 30 min to complete decolourize the methyl orange solution (Fig. 3). The catalytic performance of *in situ* formed Cu (from waste-Cu_2O) was compared with the commercial Cu powder under identical reaction conditions and was found to be almost the same (Fig. 4b-c). After completion of the reaction, the catalyst was separated by centrifugation at 3000 rpm for 30 min and the supernatant was discarded. The recovered catalyst was washed by dispersing it in 2 mL methanol and again separated by centrifugation and air-dried. In the PXRD spectrum of catalyst, after the reaction, a characteristic 2θ peak at 42.4º (111), 50.5º (200) and 74.2º (220) corresponding to the copper Miller plane was observed. Moreover, a low-intensity peak at 36.5º (111) and 61.6º (220) corresponding to the Cu_2O Miller plane was observed (Fig. 4d). The obtained data are in close agreement with the reported data [23]. The SEM image of *in situ* formed Cu from Cu_2O during the reaction is shown in Fig. 4e.

Recyclability of catalyst: A fresh batch of methyl orange decomposition reaction was performed to evaluate the recyclability of catalyst. The absorbance of the reaction mixture (0.3 mL reaction mixture + 2.7 mL distilled water) was measured at 466 nm at the beginning and after 30 min (Table-1). After completion of the first cycle, the reaction mixture was kept aside without disturbing for 2-2.5 h and the catalyst was settled at the bottom of beaker. Slowly, the liquid was decanted and 100 mL of fresh methyl orange solution was added. After adding 10 mg of $NaBH_4$ to the reaction mixture, it was kept on magnetic starrier with continuous stirring for 30 min and absorbance at 466 nm was measured again at the beginning and end of the reaction. The catalyst was recycled and reused up to five catalytic cycles without losing its catalytic potential (Fig. 5).

TABLE-1
CALCULATION OF THE PERCENTAGE OF METHYL ORANGE DECOMPOSITION DURING CATALYST RECYCLABILITY TEST EXPERIMENT

Catalytic cycle	Absorbance		Dye decomposition (%) = $[(A_0 - A_{30})/A_0] \times 100$
	Time (0 min)	Time (30 min)	
1	0.361	0.004	98.9
2	0.365	0.005	98.6
3	0.361	0.006	98.3
4	0.368	0.007	98.1
5	0.370	0.008	97.8

A_0 = Absorbance at 0 min; A_{30} = Absorbance at 30 min

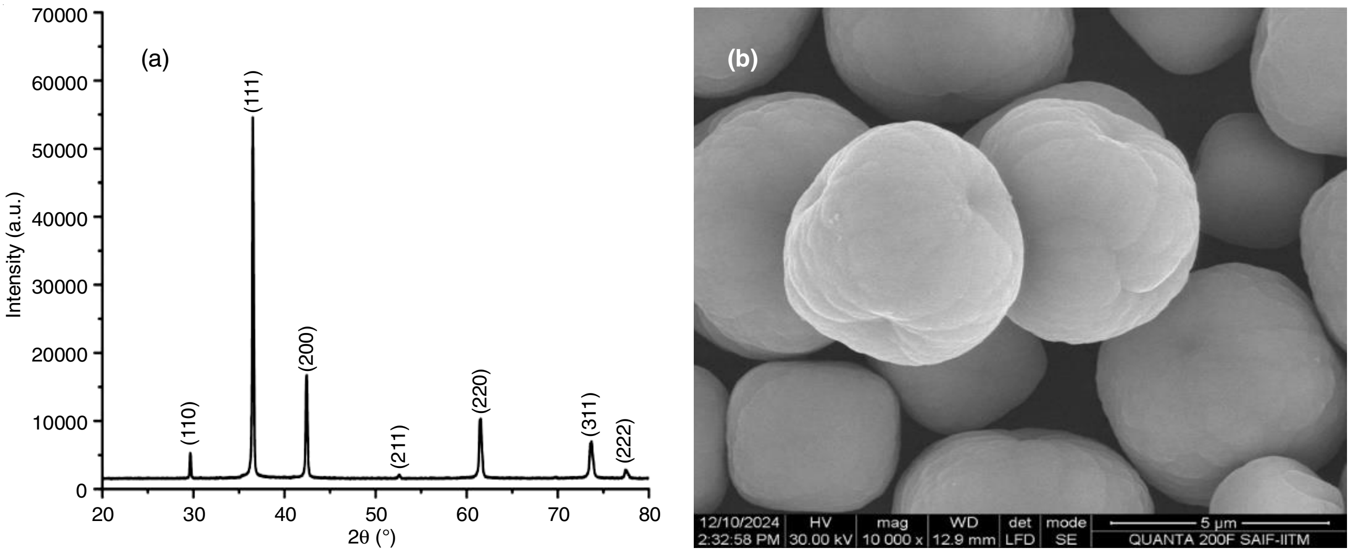


Fig. 2. Characteristic feature of Cu_2O (a) PXRD and (b) SEM image

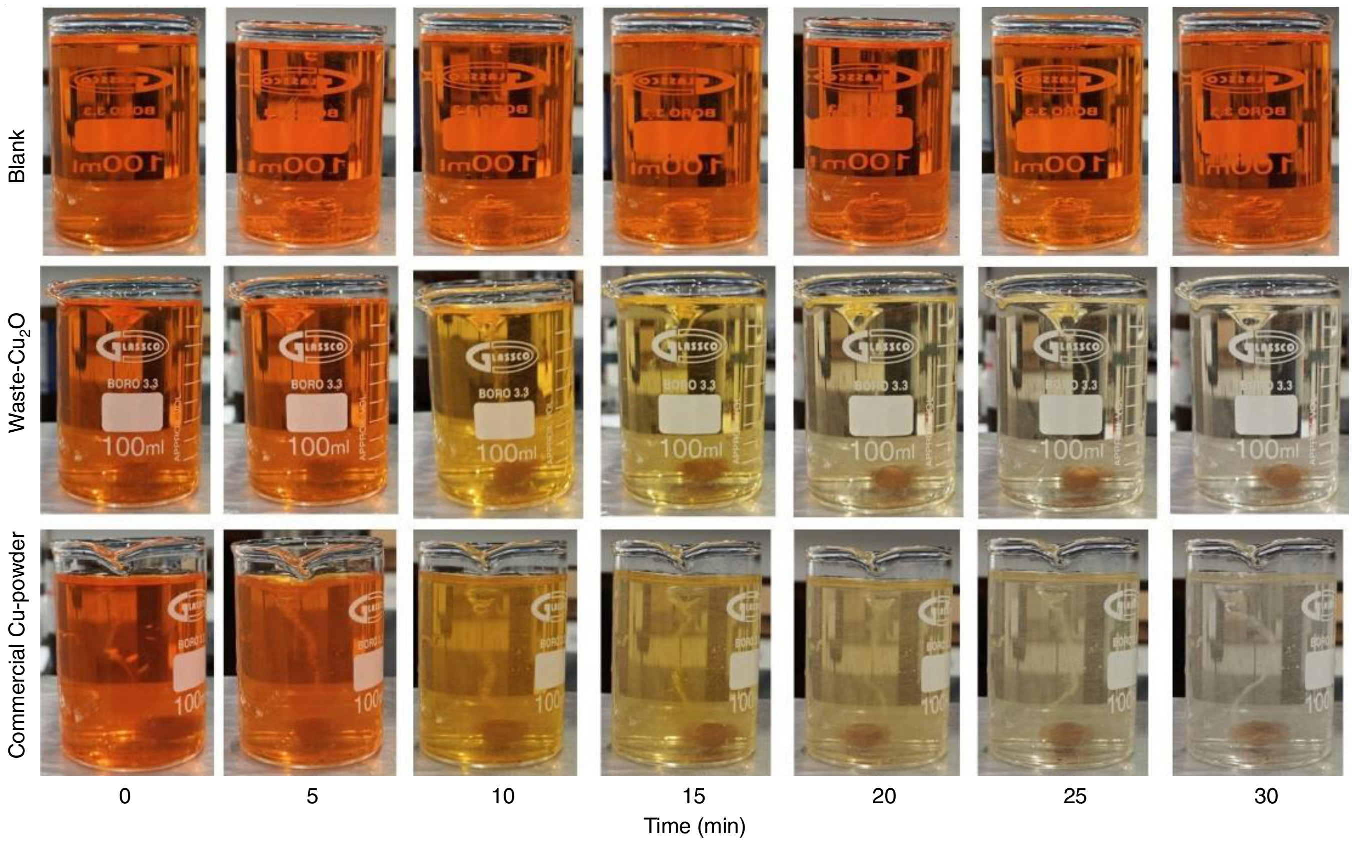


Fig. 3. Pictorial representation of reductive decomposition of methyl orange without catalyst (blank), with Cu_2O (*in situ* formed Cu) and commercial Cu-powder

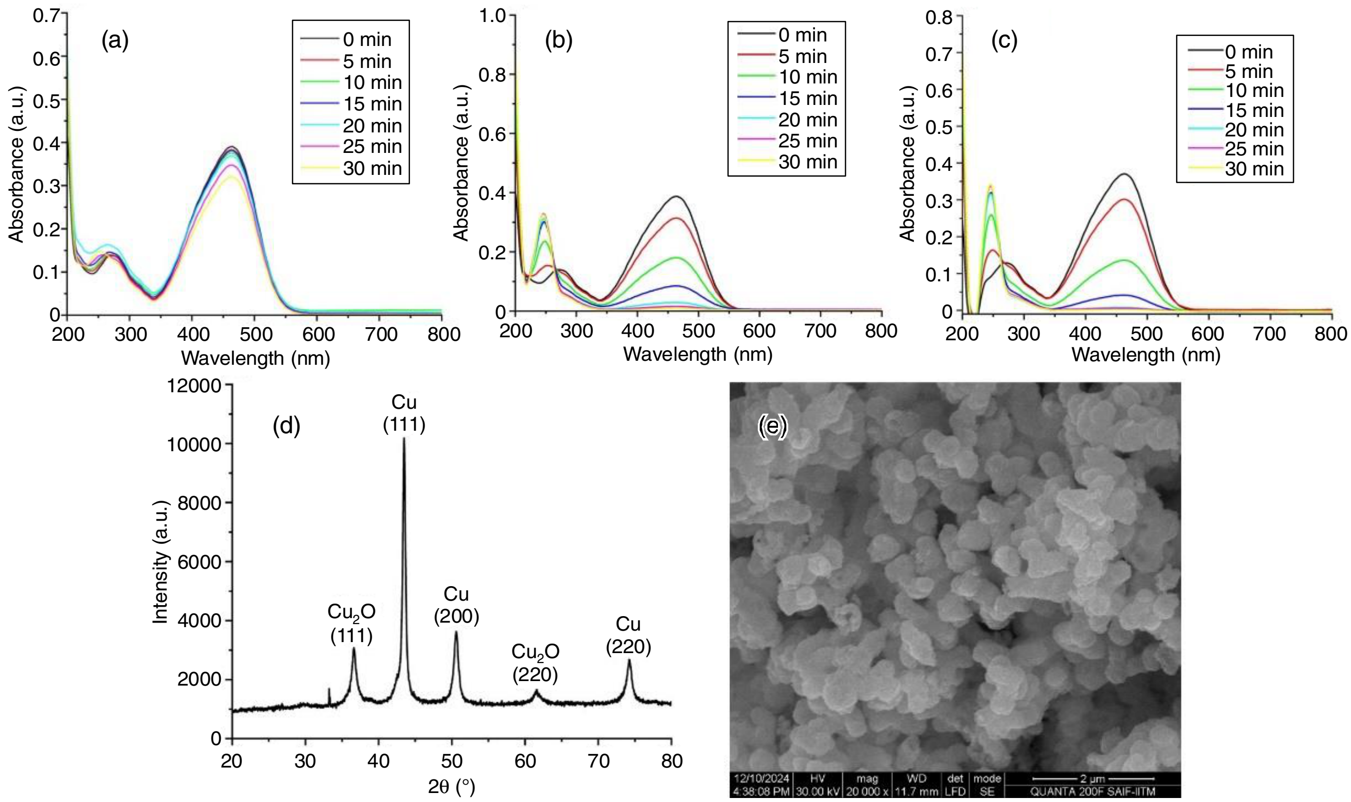


Fig. 4. UV-Vis spectra of methyl orange decomposition with and without catalyst with time: (a) blank (without catalyst), (b) waste-Cu_2O and (c) Cu-powder. Characteristic features of the waste-derived catalyst after the first catalytic cycle: (d) PXRD and (e) SEM image

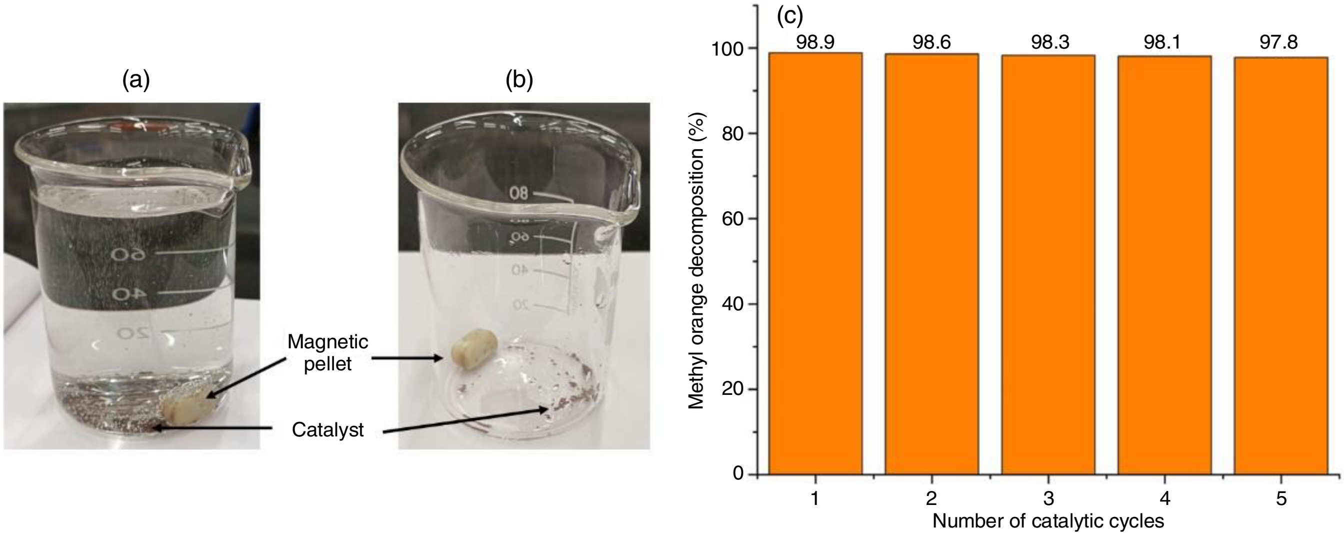


Fig. 5. (a) Image showing *in situ* formed Cu-catalyst settled at the bottom of a beaker after standing for 2 to 2.5 h, (b) recovered catalyst after decanting the water for next catalytic cycle and (c) cycling run for decomposition of methyl orange

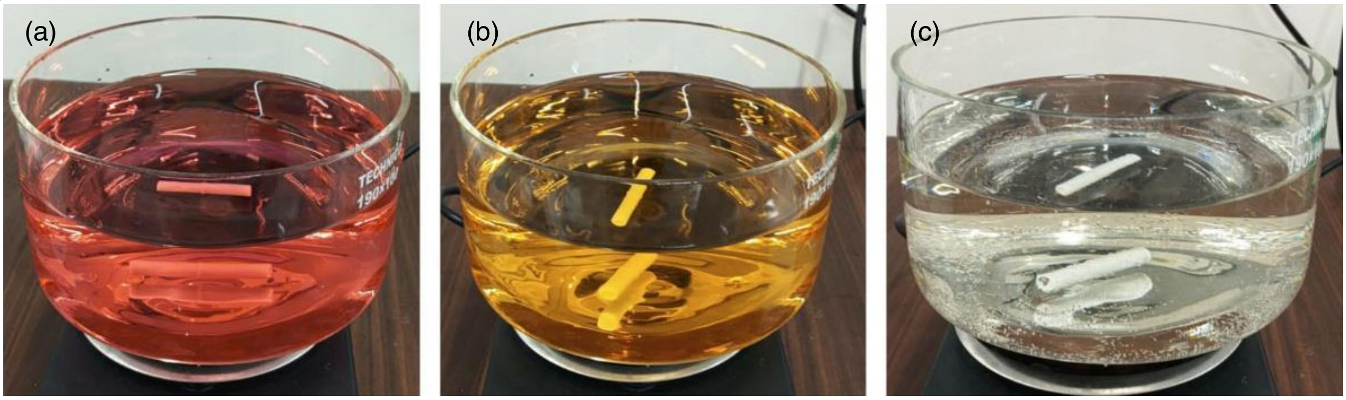


Fig. 6. Methyl orange contaminated laboratory wastewater treatment (a) wastewater (pH 3), (b) neutralized wastewater (pH 7) and (c) treated wastewater

Methyl orange contaminated laboratory wastewater treatment: The methyl orange-contaminated laboratory wastewater was treated to explore the real-life application of the isolated copper-based waste. The wastewater was generated during the practical sessions, where the students performed an acid-base titration reaction using methyl orange as an indicator. The pH of the wastewater was analyzed using standard pH paper. It was found to be around 3 and before treatment, it was neutralized (pH 7) by addition of sodium carbonate. The waste-Cu_2O (50 mg) and $NaBH_4$ (100 mg) were used against 1.5 L of wastewater. Within a minute, the complete decoration of the wastewater was observed (Fig. 6). In the UV-visible spectrum, no absorption peaks corresponding to methyl orange were observed (Fig. 7). Utilizing waste as a resource in academia is strongly advocated for resource conservation, as it will assist the younger generation in advancing sustainability and implementing a circular economy along with the principles of reduce, reuse and recycle (3Rs) whenever feasible.

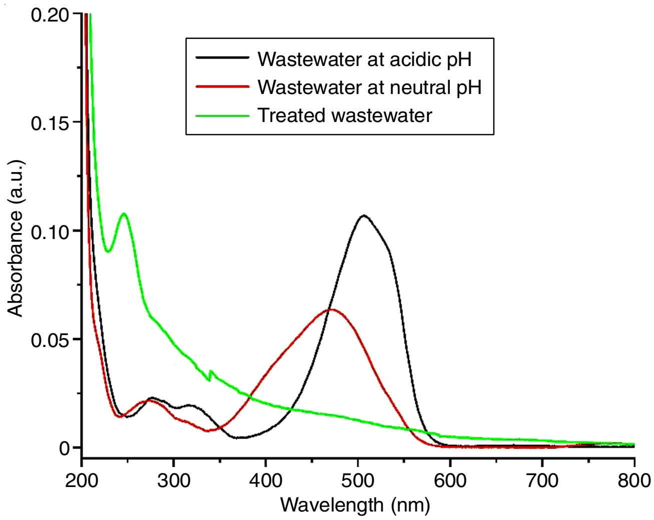


Fig. 7. UV-visible spectra of wastewater and treated wastewater

Recycling of waste-Cu_2O to Benedict's reagent: To support the circular chemical economy, the waste-Cu_2O generated from Benedict's reaction during the education process has been recycled back to Benedict's reagent. Waste-Cu_2O was first treated in an aqueous medium with H_2O_2 and H_2SO_4 to convert Cu_2O to $CuSO_4$ by following reported procedure with slight modification [18]. H_2O_2 oxidized Cu_2O to CuO. Following this, a displacement reaction takes place between CuO and H_2SO_4, resulting in the formation of $CuSO_4$ solution. An aqueous solution containing Na_2CO_3 and sodium citrate was added to the solution of $CuSO_4$ and an alkaline solution of copper citrate complex (Benedict's reagent) was obtained (Fig. 8; **Scheme-I**). Further, a test experiment was performed using the recycled Benedict's reagent with glucose (reducing sugar). A 2 mL of glucose solution was taken in a test tube and 1 mL of recycled Benedict's reagents was added and heated in a hot water bath (90 ºC),

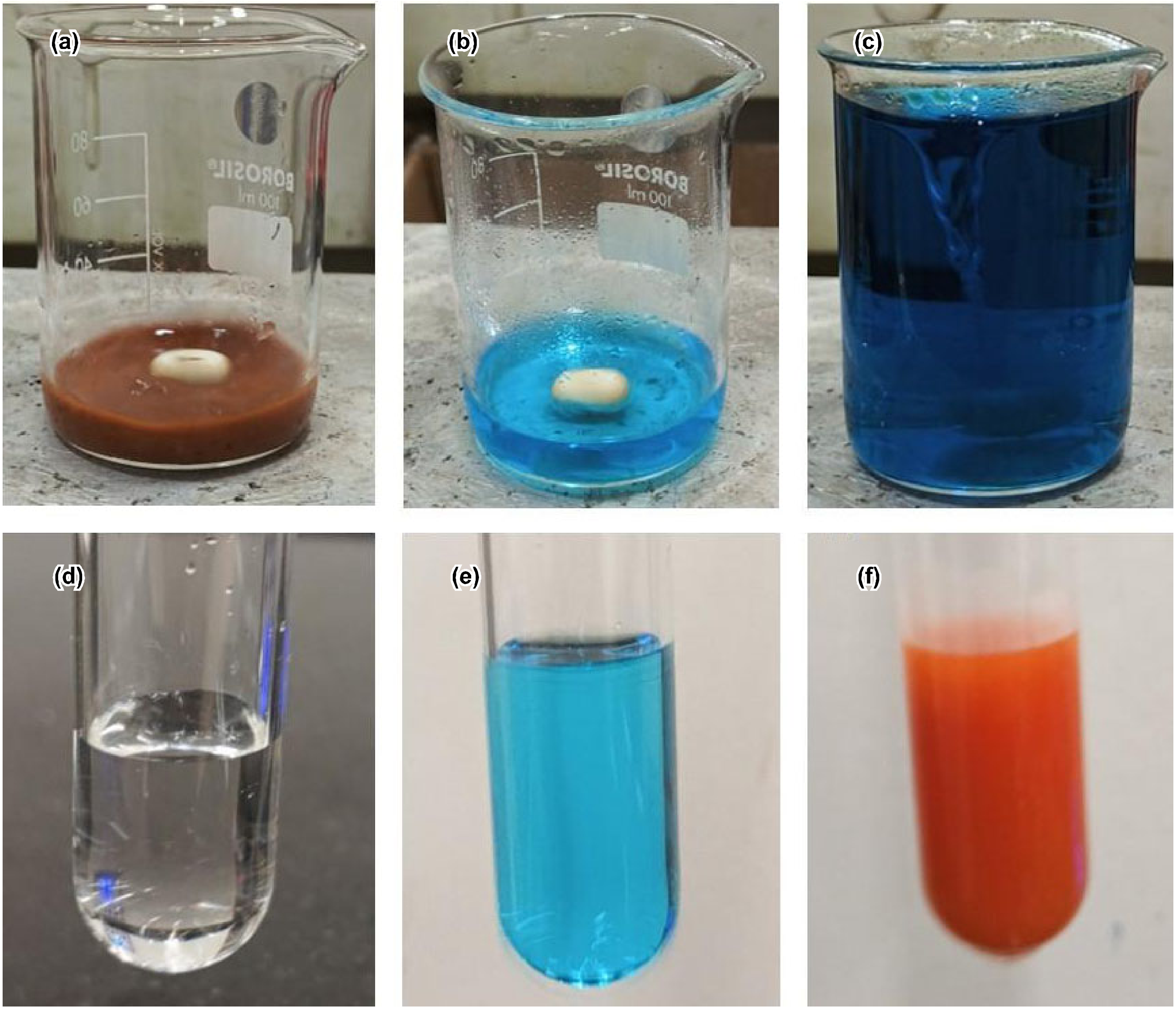
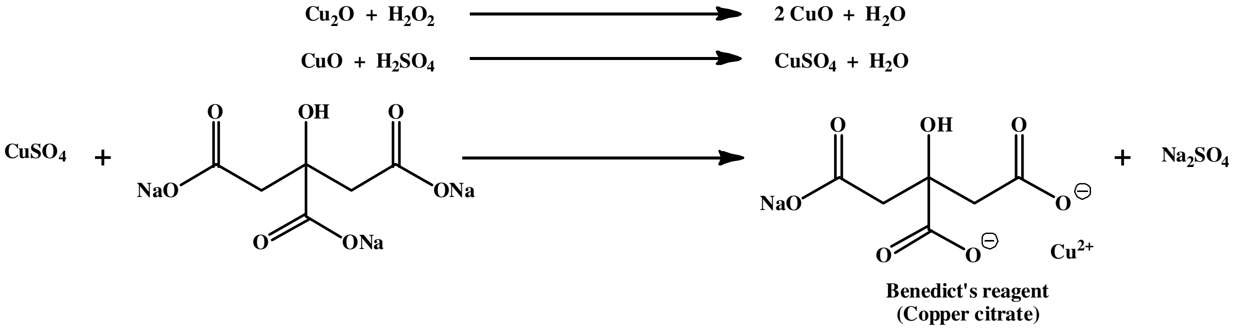


Fig. 8. Recycling of Benedict's reaction waste to Benedict's reagents (a) waste-Cu_2O and H_2O_2 in 10 mL water, (b) $CuSO_4$ solution after adding H_2SO_4 to "a," (c) Benedict's reagent, after adding a solution of sodium carbonate and sodium citrate to "b." Benedict's test using recycled Benedict's reagent, (d) glucose solution, (e) glucose solution containing recycles Benedict's reagent and (f) Positive Benedict's reaction test after heating in a hot water bath



$$Cu_2O + H_2O_2 \longrightarrow 2\,CuO + H_2O$$

$$CuO + H_2SO_4 \longrightarrow CuSO_4 + H_2O$$

Scheme-I: Stepwise schematic representation of Benedict's reaction waste recycling to Benedict's reagent within 30 sec, formation of an orange colour Cu_2O was observed (Fig. 7d,e,f). This confirmed that the waste-Cu_2O from Benedict's reagents can be recycled and reused multiple times to its maximum possible number of cycles.

Conclusion

In this study, Cu_2O was isolated from Benedict's reaction waste by following the path of a circular chemical economy. It is used as a catalyst for the reductive decomposition of methyl orange and treatment of laboratory wastewater contaminated with methyl orange using $NaBH_4$ as a reducing agent. The catalyst was recycled and reused for the five catalytic cycles. Furthermore, the waste-Cu_2O from Benedict's reaction recycled back to functional Benedict's reagent by chemical treatment. The recycled Benedict's reagent showed a positive test when an experiment was performed against a reducing sugar. Such recycling and reusing practices will help to promote green and sustainable laboratory waste management and chemical circular economy in academia.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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