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Novel Synthesis of rGO/CuO Nanocomposite and its Catalytic Application for the Oxidation of Styrene

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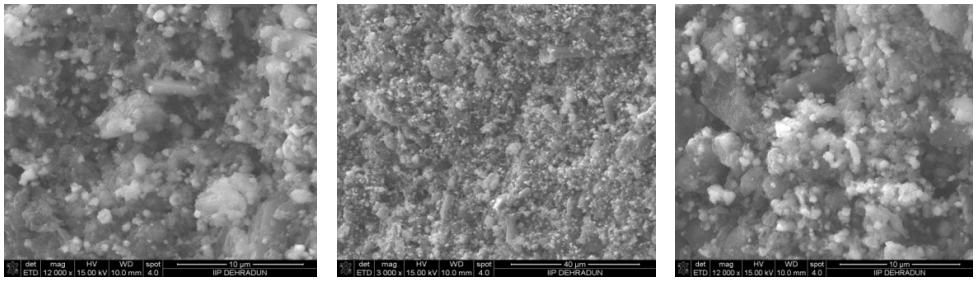
ABSTRACT

It is providing a new hydrothermal technique for creating a heterogeneous composite catalyst called reduced graphene oxide/copper oxide (rGO/CuO) nanocomposite. In order to immobilise CuO nanoparticles on surface-functionalized reduced graphene oxide sheets, the synthesis entails reducing graphene oxide (GO) and $CuCl_2$ using sodium borohydride ($NaBH_4$) acting as a reducing agent. The resultant rGO/CuO nanocomposite is a great nano-catalyst for the oxidation of unsaturated hydrocarbons like styrene because of its high catalytic efficiency, affordability, reusability, and environmental friendliness. Several analytical methods, such as Field Emission Scanning Electron Microscopy (FESEM), Energy Dispersive X-ray Spectroscopy (EDX), Thermogravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR), and X-ray Diffraction (XRD), are used to characterise the synthesized GO and rGO/CuO nanocomposites. The research shows that rGO/CuO has the potential to be a powerful catalyst in organic transformations, especially oxidation processes.

Graphical abstract:



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SEM interpretation to show adsoption of CuO on the surface of reduced graphene oxide.

Keywords: rGO/CuO nanocomposite, heterogeneous catalyst, hydrothermal synthesis, oxidation, styrene.

INTRODUCTION

Over the past two decades, designing and synthesizing catalysts with enhanced catalytic performance has emerged as a significant challenge for researchers. Nanoparticles, characterized by their nanoscale dimensions, have been considered promising catalysts due to their high surface-to-volume ratio. However, their inherent tendency to agglomerate necessitates the use of stabilizers. Concurrently, the advent of graphene and its derivatives, such as graphene oxide (GO) and reduced graphene oxide (rGO), has garnered substantial attention due to their exceptional electrical [1-2], thermal [3] and mechanical properties [1]. Moreover, graphene materials exhibit remarkable charge carrier mobility, extensive surface area, excellent biocompatibility, and high adsorption capacity [4-8].

Graphene derivatives, particularly graphene oxide, possess a large surface area enriched with functional groups, making them valuable candidates in catalytic research. Composite catalysts incorporating graphene and its derivatives are of particular interest, wherein graphene serves as a conductive substrate anchoring additional catalytic components, often metal nanoparticles. This integration stabilizes the nanoparticles without the need for external stabilizing agents. Graphene oxide/metal nanocomposites, graphene oxide/metal oxide nanocomposites, reduced graphene oxide/metal nanocomposites, and reduced graphene oxide/metal oxide nanocomposites play pivotal roles in various research domains. These include catalysis [9], gas sensing [10], biosensing [11], photocatalysis [12], organic synthesis reduction reactions [9], toxic metal ion removal [13-14], cellular imaging [15-16], drug delivery [17, 18], and other advanced applications [19-22].

Copper, a cost-effective metal, exhibits a broad spectrum of catalytic activities, ranging from C–C coupling reactions **[23]** to the synthesis of heterocycles [23]. This versatility further underscores the utility of copper-based catalysts in conjunction with graphene derivatives for advancing catalytic research.Thenanocomposite structures comprising reduced graphene oxide (rGO) integrated with CuO, Cu, and Cu_2O nanoparticles—designated as rGO/CuO, rGO/Cu, and rGO/Cu_2O, respectively—represent a class of composite catalysts wherein CuO, Cu, and Cu_2O nanoparticles are anchored onto the rGO surface. Over the past few years, numerous composite structures of this type have been synthesized for diverse applications.

For instance, copper oxide/reduced graphene oxide nanocomposites have been utilized as catalysts for synthesizing hybrid molecules containing flavanone and triazole by merging the Michael addition and Click reactions [24]. The rGO/CuOnanocomposites demonstrate efficient catalytic activity in the reduction of 4-nitrophenol to 4-aminophenol in the presence of $NaBH_4$ [9]. Additionally, rGO/CuOnanocatalysts exhibit high catalytic performance in CO oxidation reactions [25]. The Cu_2O/CuO/rGO composite has been identified as a highly effective material for supercapacitor applications [26].

Furthermore, synthesized rGO/CuO nanocomposites function as excellent photocatalysts for the degradation of organic pollutants in water and wastewater treatment [27]. These nanocomposites are more effective catalysts for visible light-induced photocatalytic degradation of pollutants such as 2-nitrophenol and 4-nitrophenol compared to bare CuO [28]. Under UV irradiation and in the presence of H_2O_2, CuO/rGO nanocomposites (50 mg) exhibit exceptional efficiency in degrading Congo red dye [39].

The CuO–Ag/rGOnanocomposite has also been identified as an efficient photocatalyst for degrading naphthol black blue dye [30]. Similarly, the rGO/Cu nanocomposite has been synthesized as a heterogeneous catalyst for Cu-catalyzedformylation and amination of aryl boronic acids [20]. Moreover, rGO/CuOnanocomposites have been developed as heterogeneous catalysts for synthesizing biologically active 1,5-benzodiazepines through the sequential addition of acyl chlorides, terminal alkynes, and o-phenylenediamines under palladium, ligand, and solvent-free conditions [31]. CuO/GO

catalysts have also been employed for the photodegradation of organic pollutants in wastewater under ambient conditions [32].

Inspired by these findings, we have undertaken the synthesis of reduced graphene oxide/copper oxide (rGO/CuO) nanocomposites as heterogeneous catalysts for the oxidation of unsaturated hydrocarbon styrene.

MATERIALS AND METHODS

Material and Methods: Graphite, concentrated sulfuric acid (H_2SO_4), potassium permanganate ($KMnO_4$), ascorbic acid, hydrochloric acid (HCl), 30% hydrogen peroxide (H_2O_2), acetonitrile (ACN), copper(II) chloride ($CuCl_2$), sodium borohydride ($NaBH_4$), and styrene were procured from LobaChemiePvt. Ltd.

General procedure:

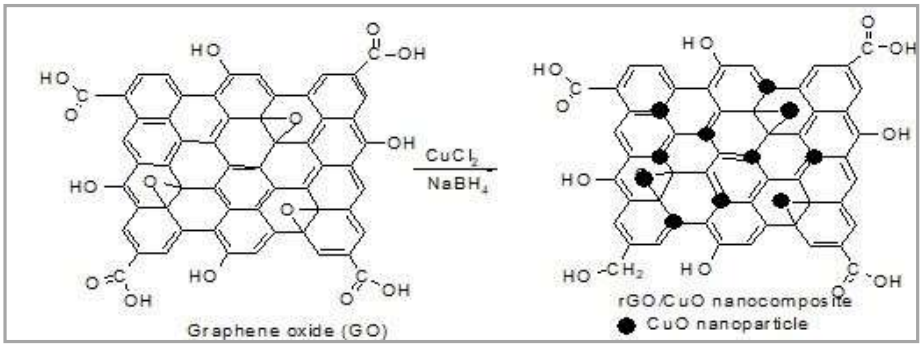
Synthesis of GO from Graphite: Graphene oxide (GO) was synthesized from graphite powder using the Modified Hummers method. In this process, 5 g of graphite powder was placed in a reaction vessel, and 7.5 mL of concentrated $H_2SO_4$was gradually added while maintaining the temperature at 0–5°C. Subsequently, 15g of $KMnO_4$ was incrementally incorporated into the mixture under continuous stirring. The reaction mixture was stirred for 2 h at 5 ± 1°C. The reaction mixture was then allowed to reach room temperature while stirring continuously for 30 min. To elevate the reaction temperature to 98°C, 200 mL of deionized water was added, and the mixture was stirred for an additional 45 min. Following this, 140 mL of deionized water and 20 mL of 30% H_2O_2were introduced into the mixture, leading to the formation of a yellow-brown precipitate, identified as graphene oxide. The precipitate was collected through vacuum filtration and washed with 100 mL of 5% aqueous HCl. The resulting solid material was dried at 60°C in a vacuum oven [33-35].

Preparation of rGO/CuOnanocomposite: A suspension comprising graphene oxide (GO, 25 mg), $CuCl_2$ (15 mg), and deionized water (250 mL) was prepared in a 500 mL round-bottom flask. The mixture was subjected to ultrasonication at 50 Hz for 1.5 h at room temperature. Subsequently, a 1% NaBH4 solution (10 mL) was added dropwise, and the reaction mixture was stirred at 80°C for 18 h. Upon completion, the reaction mixture was cooled to room temperature, and the reduced graphene oxide/CuO (rGO/CuO) nanocomposites were isolated via centrifugation. The obtained rGO/CuO nanocomposites were dried at 100°C and subsequently characterized using various analytical techniques, including X-ray diffraction (XRD), energy-dispersive X-ray spectroscopy (EDX), thermogravimetric analysis (TGA), field-emission scanning electron microscopy (FE-SEM), Fourier-transform infrared spectroscopy (FTIR), and UV-Visible spectroscopy. The product was designated as the rGO/CuO nanocomposite.

Detection Method: The rGO/CuO nanocomposite synthesized via the hydrothermal method was characterized using a range of analytical techniques. Fourier-transform infrared (FTIR) spectroscopy was conducted using a Thermo Scientific instrument to analyze the functional groups present. X-ray diffraction (XRD), an essential technique for determining material properties, was performed using a Bruker AXS D8 Advance A25-X1-1A2Z2C4B0 diffractometer to obtain the catalyst's XRD pattern. Scanning electron microscopy (SEM) was employed to investigate the composite's morphology, utilizing a field-emission SEM (FE-SEM) instrument (model: JSM-7610F-Plus) in conjunction with an Au sputter coater (model: DII-29030SCRT) to coat samples with gold prior to analysis. Thermogravimetric analysis (TGA) was performed to assess the thermal stability of the nanocomposite using a PerkinElmer thermal analyzer. These comprehensive characterizations provide insights into the structural and thermal properties of the synthesized rGO/CuO nanocomposite.

RESULTS AND DISCUSSION

Synthesis of rGO/CuO nanocomposites: rGO/CuO nanocomposite was prepared through the instantaneous chemical reduction of graphene oxide (GO) to reduced graphene oxide (rGO) using sodium borohydride ($NaBH_4$) and the simultaneous conversion of $CuCl_2$ to CuO nanoparticles (Scheme 1).



Scheme 1. Synthesise of rGO/CuO.

SEM Interpretation: The SEM images were attained at magnifications of 10 to 50 µm at figure 1. It is apparent that the morphology of intercalated nanocomposite particles is truly in nanosize and it shows the uncoordinated morphology due to the agglomeration of particles in the solution while synthesis. The amalgamation between CuO and rGo surface is a assortment of interfaces such as hydrogen bonding, vanderwalls forces and electrostatic interactions [36, 37]. Outcomes reveal that there are two types of morphologies: spherical form CuO and wrinkled sheet-like form of rGo, indicating that CuO NPs are firmly affixed to the surface of these sheets. On the whole, SEM results showed the homogeneous distribution of CuO over rGo's rumpled, paper-like surface as seen in SEM illustrations of rGO/CuO, indicating that CuO nanoparticles are incorporated into rGo.

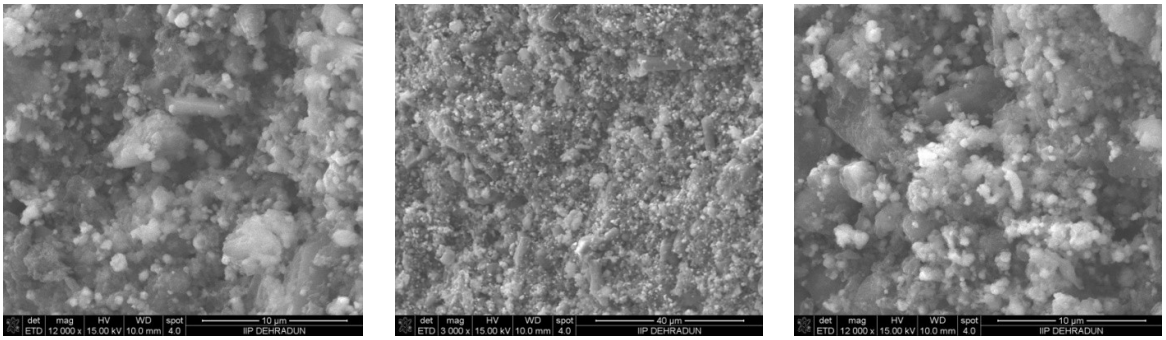


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Figure 1. SEM interpretation to show adsoption of CuO on the surface of reduced graphene oxide.

EDX Interpretation: The EDX mapping is used to examine the structure of the prepared nanocomposite. The EDX spectrum of prepared rGO/CuO nanocomposite shows the presence of 54.68% carbon, 19.63% oxygen, and 25.57% copper which confirms the rGO surface coated with CuO nanoparticles as well as the successful synthesis of rGO/CuO nanocomposite [9, 28].

XRD Interpretation: The powder X-ray diffraction (PXRD) pattern of GO shows the characteristic diffraction peak at 2θ ~10.3 and 42.8 for [001] plane due to oxygen containing functional groups on carbon skeleton and [100] planes due to hexagonal carbon skeleton respectively indicating the

successful oxidation of graphite into graphene oxide (GO). In the PXRD pattern of synthesized rGO/CuO nanocomposite structure disappearance of the [100] plane at 2θ~42.8 and concomitant appearance of a peak at 2θ ~23.15 indicating the reduction of GO into rGO during the decoration of CuO nanoparticles onto GO. The XRD pattern of synthesized rGO/CuOnanocomposite shows various small distinct diffraction peaks at 2θ ~26.55, 28.2, 32.85, 36.04, 39.1, 50.58 and 54.07 confirm the deposition of CuO nanoparticles over the surface rGO successfully [**30-39**].

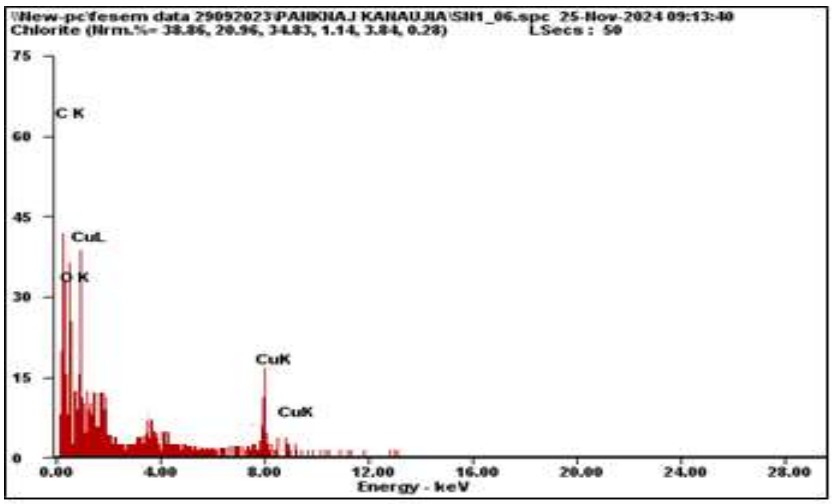


Figure 2. EDX data to show successful synthesis of rGO/CuO nanocomposite.

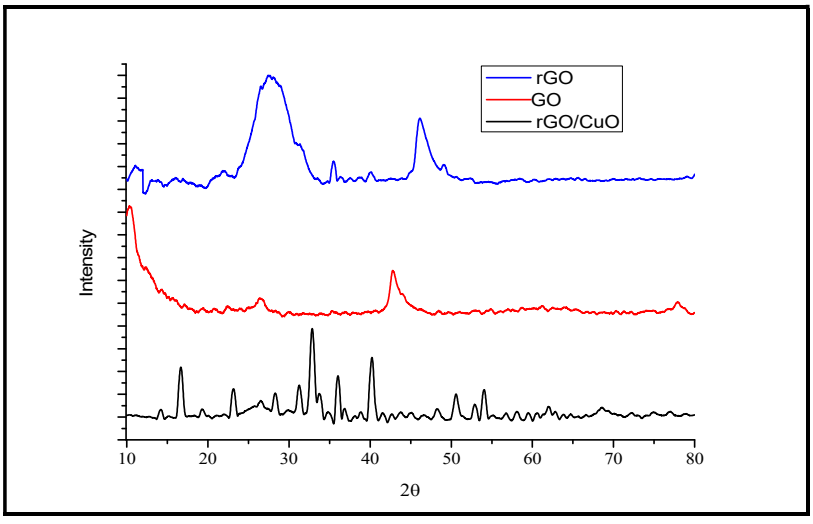


Figure 3. XRD spectra to show CuO crystal present on the surface of rGO.

TGA Interpretation: Thermogravimetric analysis (TGA) is a technique employed to assess the thermal stability of a compound. The TGA of rGO/CuO nanocomposite exhibits a mass loss of approximately 12.43% below 100°C, attributed to the elimination of moisture and water content. Between 100–400°C, a mass loss of around 6% occurs due to the degradation of oxygen-related functionalities, with additional mass loss occurring above 400°C, linked to the pyrolysis of the carbon skeleton. These TGA observations are consistent with findings from previous studies [**40**].

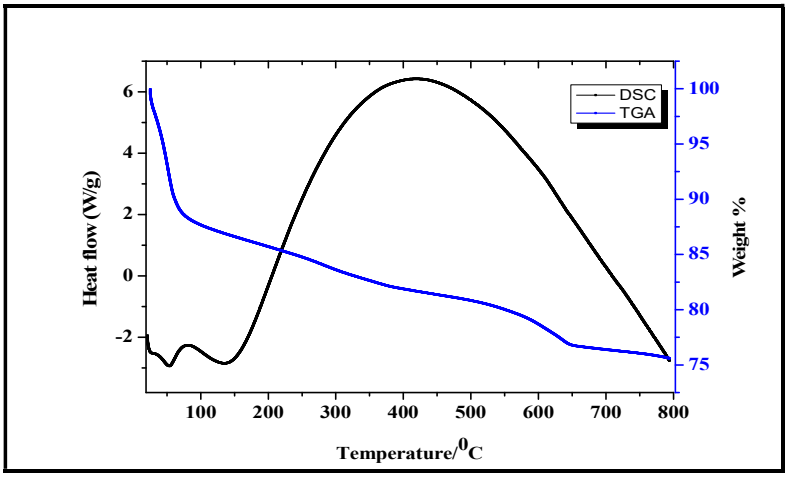


Figure 4. TGA and DSC spectra to show stability of rGO/CuO.

FTIR Interpretation: The FTIR spectra of rGO/CuO nanocomposite showing the peaks at 1622 cm^{-1}, 1384 cm^{-1} and 1012 cm^{-1} corresponding to H-O-H bending vibration, =C-H vibration and peak corresponding to CuO nanoparticles with decreased intensity with respect to GO, which indicates the reduction of GO during the synthesis of rGO/CuO nanocomposite [38]. Peak appeared at 3431 cm^{-1} indicating O–H stretching vibration, which gets significantly reduced during the synthesis of rGO/CuO from GO [41]. Peak appeared at 459cm^{-1} and 595 cm^{-1} are due to Cu-O streatchingvibration, indicating the decoration of CuO onto the rGO surface [9, 41].

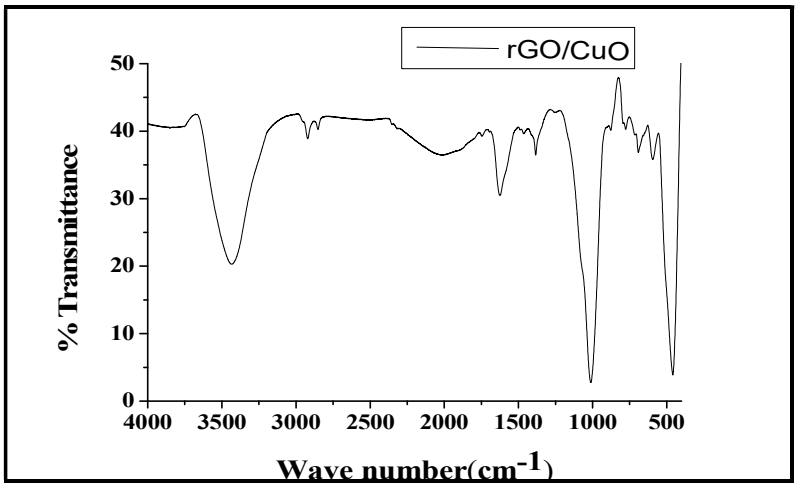
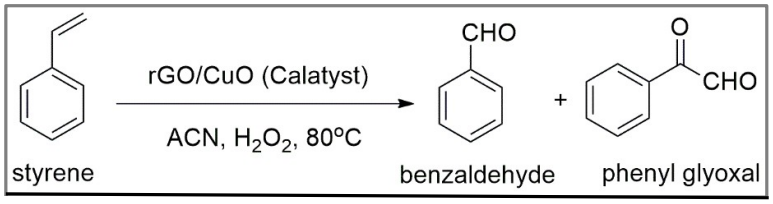


Figure 5. FTIR spectrum of rGO/ CuO.

Catalytic activity of rGO / CuO nanocomposite: The synthesized rGO/CuO nanocomposite was employed as a heterogeneous catalyst in the oxidation of the substrate (styrene) using hydrogen peroxide (H_2O_2) as the oxidizing agent. Initially, 10 mmol of styrene (substrate) was introduced into a round-bottom flask along with 15 mg of the synthesized rGO/CuO nanocomposite (catalyst) and 15 mmol of hydrogen peroxide (H_2O_2) in 10 mL of acetonitrile solvent. The flask was connected to a reflux condenser and placed in a heating mantle at 80 °C for 6 hours. Under these conditions, catalytic oxidation of styrene occurred, yielding benzaldehyde as the major product with a 61% yield and phenyl glyoxal as the minor product with a 6% yield, as illustrated in the following reaction (Scheme 2).



Scheme 2..rGO/CuOcatalyzed oxidation of styrene.

Test for recyclability and heterogeneity of the reaction: The recyclability of rGO/CuO nano composite tested for the oxidation of styrene under the optimized condition after separating the used catalyst from the reaction mixture, washing with acetonitrile and drying at 120°C. The first cycle of experiment gives 61 % while the second cycle gave 58% conversion of styrene into benzaldehyde. To check the heterogeneous nature of catalyst we stoped reaction after 3 h and removed the catalyst by filtration. The filtrate collected after separating the solid catalyst was continued for another 3 h. The GCMS analysis showed no further increment in the conversion. This confirms that the reaction did not proceed upon removal of catalyst. The resulted data should be well presented by the form of schemes, figures, graphs, tables, reactions and equations. These items should be numbered clearly.

APPLICATION

The rGO/CuO nanocomposite exhibits potential as a catalyst for diverse oxidation reactions involving unsaturated hydrocarbons. Additionally, this catalyst is being investigated for its efficacy in the degradation of dyes. The ongoing study has yielded promising results.

CONCLUSION

We report the synthesis, characterization, and application of reduced graphene oxide rGO/CuO nanocomposites for the catalytic oxidation of styrene. The CuO nanoparticles were synthesized in situ and anchored onto surface-functionalized rGO sheets, resulting in a nanocomposite with promising catalytic properties. The rGO/CuO nanocomposite exhibited good catalytic activity for the oxidation of styrene. Furthermore, the catalyst demonstrated excellent recyclability and heterogeneous nature, highlighting its potential as a sustainable and cost-effective material in the framework of green chemistry.

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Authors Contribution: All Authors are equally contributed in preparing the manuscript.

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