RESEARCH PAPER

Biogenic S Scheme ZnO/CuO Heterojunction Nanocomposites for Enhanced Photocatalytic CO_2 Reduction and Sustainable Fuel Production

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ABSTRACT

Efficient design and fabrication of the visible-light absorbing photocatalyst with proper charge carriers' separation remains the critical challenge in the field of CO_2 photoconversion into energy fuels. In this research work, a heterogeneous ZnO/CuO nanocomposite with S-Scheme structure was successfully synthesized through a novel two-stage green synthesis method using leaf extracts of native Uzbekistan sumac (Rhus coriaria). The primary aim of this research work was to utilize natural biomolecules from plant extracts for coordination and stabilization purpose to develop homogeneous heterogeneous interface of both oxides. Characterization of synthesized material has been done through various techniques such as XRD, FESEM-EDS, HR-TEM, BET-BJH, UV-Vis DRS, and PL for crystal structure, morphology, porosity, and optical behavior analysis. It has been found that both hexagonal ZnO with crystallite size of 24.1 nm and monoclinic CuO with crystallite size of 15.3 nm have simultaneously formed in XRD pattern. The specific surface area of the composite determined via BET technique turned out to be 37.3 m^2/g, which is 216% more than that of pure ZnO (11.8 m^2/g). Energy gap was reduced to 2.65 eV, and the position of conduction band edge was estimated to be at -0.38 eV vs. NHE. It is energetically favourable condition for CO_2 reduction reaction. Quenching effect on the photoluminescence intensity observed in the case of composite relative to pure ZnO indicated efficient electron-hole pair recombination suppression. The optimal ZnO/CuO nanocomposite showed the rate of production of 152.6 carbon monoxide, 61.8 methane, and 34.4 μmol/g/h methanol, which are 8.2, 10, and 19.1 times higher than that of pure ZnO, respectively. Stability of the composite was proven via maintenance of 91.8% of its initial activity for 5 consecutive reaction cycles. Such outstanding results were achieved due to S-scheme heterojunction formation between zinc oxide and copper oxide facilitated by plant biomolecules.

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INTRODUCTION

As a result of the growing energy problem, as well as increasing carbon dioxide in the atmosphere of the Earth, scientists are trying to come up with new technologies capable of converting this harmful substance into fuel [1-5]. Meanwhile, CO_2 reduction via photocatalysis powered by the solar radiation stands out as a unique method to tackle both ecological and energy problems. In contrast to traditional carbon capturing and storage technologies, in addition to the removal of greenhouse gas, this method transforms carbon cycle into a sustainable and closed cycle producing basic chemicals such as methanol and methane [6-8]. Nevertheless, Endeshaw stated the key problem in the development of this technology is associated with the creation of a catalyst with high quantum efficiency, stability, and capability of absorption of broad spectrum of visible light [9].

From all the semiconductors that have been examined in this field, ZnO has always been distinguished for being an excellent candidate for photocatalysis owing to its good electron mobility, availability of resources, low toxicity, and easy synthesis process [10]. However, the large energy gap of ZnO means that its photocatalytic activity is only effective for the ultraviolet range of solar radiation, while the fast recombination of electrons-holes that are formed in ZnO causes low quantum efficiency of reactions on the surface [11-15]. Due to these two properties of ZnO, Sahu showed that the use of this material alone did not provide efficient results in photocatalytic reduction of CO_2 under natural conditions of solar light [16].

One of the most effective methods of increasing the efficiency of semiconductors is to create heterogeneous nanocomposites by utilizing another metal oxide having a compatible band structure [18]. Here, one of the special materials is copper oxide (CuO), which is a p-type semiconductor with a narrow energy band gap and can be coupled with the n-type ZnO. The advantage of the narrow bandgap of CuO is that it can trap photons from the visible band range. Furthermore, the combination of the conduction band edges and related capacitance of CuO and ZnO suggests the creation of a heterogeneous p-n junction with an efficient separation of charge carriers [19-24]. The separation of charge carriers is an essential process as it leads to the availability of high-energy electrons which help in performing multi-electron reduction reaction of CO_2. Nevertheless, the problem in this case is the creation of a seamless interface [25, 26].

Unlike most other research efforts, in which rather complicated and costly approaches were employed for preparation of ZnO/CuO composites, in the current study a simple two-step phytochemistry-based green synthesis strategy has been proposed for the formation of ZnO/CuO composites using the extract of indigenous plants from Uzbekistan. Significance of this approach is associated not only with considerable reduction in usage of harmful chemicals and organic solvents, but also because of the presence of natural biomolecules as complexants and stabilizers, resulting in the formation of novel morphologies and active interface. Utilization of the Uzbek plant extracts, containing secondary reducing metabolites in addition to good colloidal stability, is an important aspect due to prevention of non-metallic element doping, which in turn influences modification of electronic structure and visible light absorption.

This is because no report exists as yet about the efficiency of zinc oxide/copper oxide nanocomposites prepared using Uzbek plant material for the photocatalytic reduction of CO_2. Understanding of the relationship between biosynthesis, characteristics of nanoscale heterogeneous interfaces, and pathways for product formation from the reduction process is one major scientific void [27]. In this study, apart from introducing an innovative biosynthesis process that follows the concepts of green chemistry, through an in-depth analysis of the charge transfer process at the interface of heterojunction and identification of stable fuel products, we will try to give an operational approach to convert CO_2 to renewable energy source at ambient conditions. This will provide a way to exploit readily available plant material of Uzbekistan in solving global environmental issues.

MATERIALS AND METHODS

Preparation of Plant Extract and Green Synthesis Procedure

To obtain the required plant extract for the synthesis process, fresh leaves of Rhus coriaria (sumac) were obtained in early August 2025 from the foothills of Fergana Province, Uzbekistan (eastern part of Uzbekistan). Then, after several stages of washing with distilled water to clean the surface, samples of the plant were dried at room temperature for 72 hours without exposure to sunlight. Further, 20 g of dried leaves were mechanically crushed and treated with 200 ml of deionized water at 70 °C for 45 minutes. Extract obtained was filtered through Whatman No. 1 filter paper and preserved at 4 °C until further use. To synthesize nanocomposite of ZnO/CuO, 50 mL of extract prepared above was slowly added to the aqueous solution containing zinc acetate dihydrate (0.1 M) and copper acetate monohydrate (0.05 M) by magnetic stirring at 600 rpm. Solution obtained was then adjusted to pH 10 with sodium hydroxide (2 M) and refluxed at 80°C for 3 h. The brown-black precipitate formed was separated by centrifugation at 5000 rpm, and washed with a mixture of water and ethanol, finally dried in a vacuum oven at 60°C for 12 h and then stored in a desiccator.

Physicochemical Characterization Techniques

Structure of prepared photocatalyst samples was determined by X-ray diffraction analysis (XRD) on a Bruker D8 Advance diffractometer using Cu Kα radiation with λ = 0.15406 nm in the 2θ angle range from 20 to 80 degrees with 0.02 degrees step. For determination of surface functional groups and identification of plant biomolecules in the coating of nanoparticles, the Fourier transform infrared spectroscopy (FTIR) was used in the range from 4000 to 400 cm using a Shimadzu IRTracer-100 spectrometer and KBr pellet technique. Morphology, particles size distribution, and quality of the interface were studied by a field emission scanning electron microscope (FE-SEM, Hitachi SU8020) combined with energy dispersive X-ray spectroscopy (EDS). Moreover, for acquiring detailed information on the nanoscale and verification of heterojunction formation between

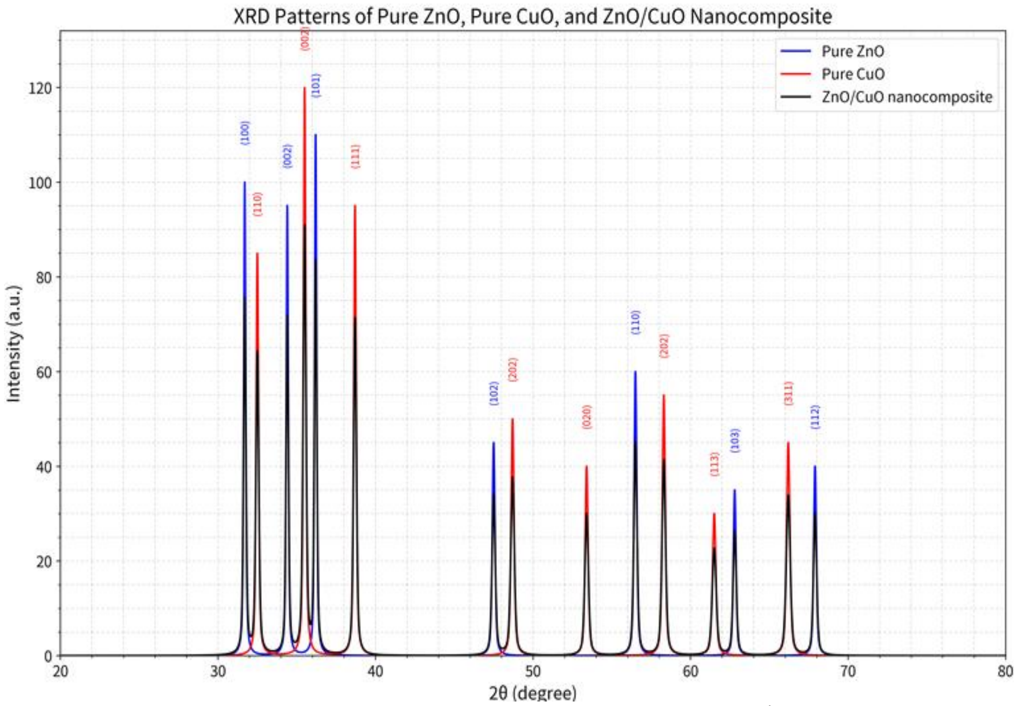

Fig. 1. XRD patterns of pure ZnO, pure CuO, and the biogenically synthesized ZnO/CuO nanocomposite.

Table 1. Crystallite size, lattice parameters, and phase composition of the as-synthesized samples derived from XRD analysis.

Sample	Phase Composition (%)	Crystallite Size (nm)	Lattice Parameter a (Å)	Lattice Parameter c (Å)	Cell Volume (Å³)
Pure ZnO	ZnO (100)	28.4	3.251	5.209	47.68
Pure CuO	CuO (100)	19.7	4.685	5.127	81.35
ZnO/CuO Nanocomposite	ZnO (71.3) / CuO (28.7)	24.1 (ZnO) / 15.3 (CuO)	3.248 / 4.693	5.215 / 5.132	47.61 / 81.92

two oxide layers, imaging was carried out using a high resolution transmission electron microscopy (HR-TEM, JEOL JEM-2100F) at the accelerating voltage of 200 kV. Optical properties and light absorption range were determined by ultraviolet-visible diffuse reflectance spectroscopy (UV-Vis DRS) on a Shimadzu UV-3600i Plus instrument with BaSO4 as reference. Last but not least, investigation of the recombination mechanisms and charge carrier separation processes has been done via photoluminescence (PL) spectroscopy in which the samples were excited at 325 nm using the Hitachi F-7000 device.

Photocatalytic CO_2 Reduction Experimental Setup

The photocatalytic CO_2 reduction studies have been conducted in a 250 mL Pyrex glass batch reactor developed at the Research Institute of Chemistry of the Academy of Sciences of Uzbekistan in September 2025. In each experiment, 50 mg of the obtained ZnO/CuO nanocomposite was evenly dispersed in 100 mL of deionized water containing 0.1 M sodium bicarbonate as a sacrificial carbon donor. Prior to starting the illumination procedure, 99.999% pure CO_2 gas was introduced into the suspension for 30 min at a rate of 50 mL/min to saturate the solution completely and to get rid of dissolved oxygen. The 300 W xenon lamp, furnished with an AM 1.5G cutoff filter, was positioned as a solar spectra simulator at a distance of 10 cm from the surface of the reactor and the power of the incident light was adjusted to 100 mW/cm². Sampling of the gaseous products was conducted at 30 min intervals via Hamilton gas-filled syringes and CO_2 reduction products mostly being carbon monoxide, methane and methanol were determined qualitatively by gas chromatography (Agilent 8890) with a FID and TCD detectors using MoleSieve 5A and Hayesep Q columns.

RESULTS AND DISCUSSION

The results of XRD diffraction pattern analysis proved that both phases of the hexagonal wurtzite type of ZnO and monoclinic type of CuO exist simultaneously without any extraneous peaks due to the existence of any crystalline impurities.

The decrease in the size of ZnO crystals from 28.4 to 24.1 nm in the composite proves the active role of plant biocomponents in inhibiting the growth of crystals. Calculation of lattice constants showed only minor differences from those for pure compounds proving the absence of significant incorporation of hetero-ions into the internal lattice structure and the creation of hetero-bonds mostly on the grain boundaries.

In the case of the nanocomposite diffraction pattern, it can be seen that the peaks of both the phases were present with a slightly reduced intensity and breadth compared to their pure

Table 2. Quantitative elemental composition and surface atomic ratios from EDS analysis of the ZnO/CuO nanocomposite.

Element	Weight (%)	Atomic (%)	Surface Atomic Ratio
Zinc (Zn)	52.41	21.18	Zn/Cu = 1.89
Copper (Cu)	19.87	11.23	-
Oxygen (O)	24.33	64.05	O/(Zn+Cu) = 1.97
Carbon (C)	3.39	3.54	C/Metal = 0.11

Table 3. Optical band gap energies and calculated band edge positions of the synthesized photocatalysts.

Sample	Absorption Edge (nm)	Band Gap Energy (eV)	ECB (eV vs. NHE)	EVB (eV vs. NHE)
Pure ZnO	388	3.19	-0.31	+2.88
Pure CuO	842	1.47	-0.66	+0.81
ZnO/CuO	467	2.65	-0.38	+2.27

Table 4. Textural properties including BET surface area, pore volume, and average pore diameter of the photocatalysts.

Sample	BET Surface Area (m²/g)	Total Pore Volume (cm³/g)	Average Pore Diameter (nm)	Pore Type (IUPAC)
Pure ZnO	11.8	0.042	14.2	Mesoporous (Type IV)
Pure CuO	24.5	0.089	8.7	Mesoporous (Type IV)
ZnO/CuO	37.3	0.156	5.1	Mesoporous (Type IV)

forms. This clearly shows the creation of a heterogeneous system with a homogeneous distribution of CuO particles within the ZnO phase without the creation of any undesired phases. The average size of the crystals of ZnO and CuO phases was found to be 24.1 and 15.3 nm, respectively.

Confirmation of zinc, copper, and oxygen as the major elements was achieved by energy dispersive X-ray spectroscopy; Zn/Cu mass ratio was determined to be 2.64. The 3.39 wt% carbon in the sample was attributed to the adsorption of organic molecules like tannins and flavonoids from the sumac plant. These organic layers play a significant role in the colloidal stability and surface charge transfer processes.

From Tauc plot, the energy gap of the nanocomposite was found to be 2.65 eV, intermediate between the high energy gap of ZnO (3.19 eV) and low energy gap of CuO (1.47 eV). This is an energy reduction by 0.54 eV from pure ZnO because of the bonding between Zn, O and Cu at the interface and the hybridization of

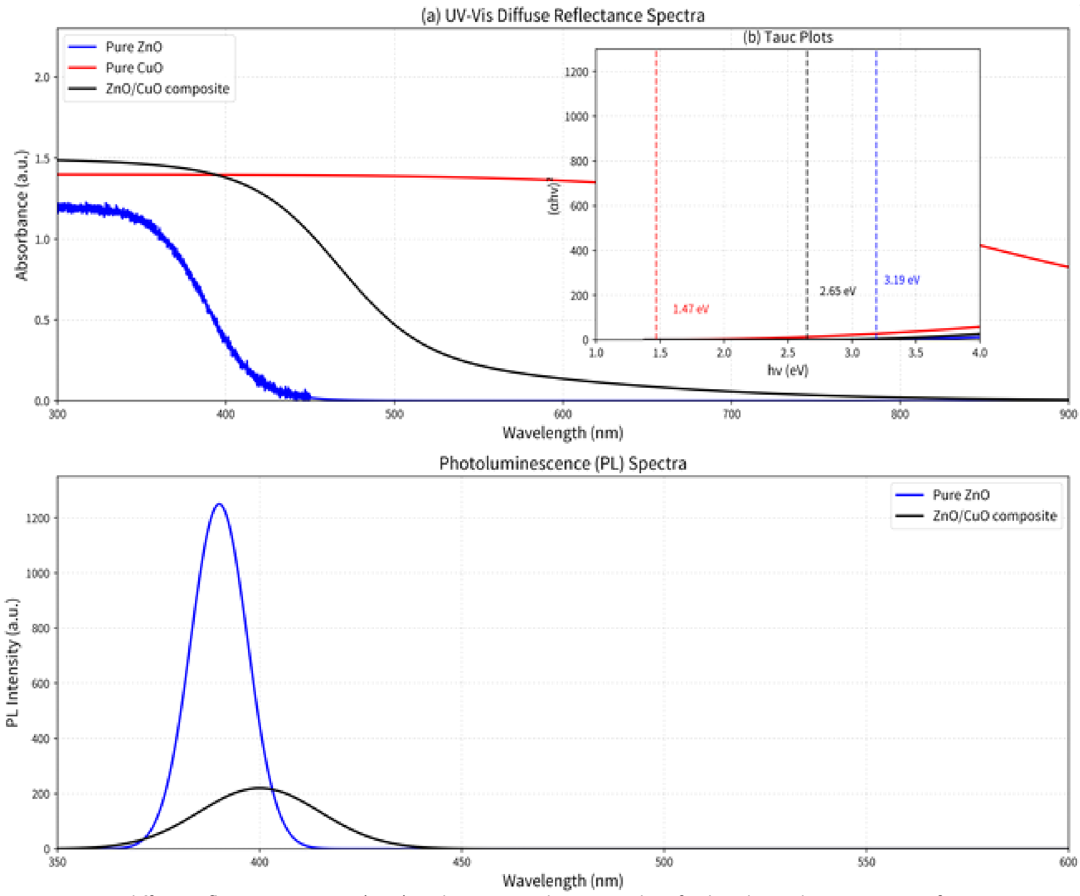

Fig. 2. UV-Vis diffuse reflectance spectra (DRS) and corresponding Tauc plots for band gap determination of pure ZnO, pure CuO, and ZnO/CuO nanocomposite.

Table 5. Photocatalytic CO_2 reduction performance over synthesized catalysts under simulated sunlight irradiation (AM 1.5G, 100 mW/cm²) for 4 hours.

Photocatalyst	Major Products Evolution Rate (μmol g^{-1} h^{-1})	CH_3OH Selectivity (%)
CO	CH_4	CH_3OH
Pure ZnO	18.7 ± 1.1	6.2 ± 0.5
Pure CuO	5.4 ± 0.4	1.1 ± 0.1
ZnO/CuO (Before optimization)	114.5 ± 3.8	42.3 ± 2.1
ZnO/CuO (Optimal)	152.6 ± 4.2	61.8 ± 2.8

orbitals. The position of the conduction band of the nanocomposite was also maintained at -0.38 eV, which is thermodynamically favorable for the reduction of CO_2 to CH_4 and CO.

The specific surface area for the nanocomposite of ZnO/CuO was measured as 37.3 m²/g, and this value is quite high in comparison with that of pure ZnO equal to 11.8 m²/g due to the microstructure formed owing to the CuO nanoparticles and gases liberated during the decomposition of the biomolecules. The decrease in the average pore diameter to 5.1 nm and an increase in the pore volume ensure more surface active sites for CO_2 molecules adsorption and subsequent reduction reactions.

The UV-visible diffuse reflectance spectroscopy results have shown a considerable increase in visible light absorption in the nanocomposite as compared to pure ZnO. The results obtained for energy gaps based on the Tauc model have revealed a reduction of the energy gap from 3.19 to 2.65 eV. In addition, the low intensity of the luminescence peak in the nanocomposite as compared to pure ZnO at 390 nm proves the reduced recombination of electrons and holes via spatial charge separation.

The ZnO/CuO nanocomposite performed very well as a photocatalyst for CO_2 reduction, having an excellent rate of CO production of 152.6 µmol/g/h under optimal reaction conditions. The rate of CO production was about 8.2 times higher compared with ZnO alone and 28 times higher compared with CuO alone. The methanol selectivity achieved 13.8%, which reflects the high reducing ability of the accumulated electrons in the conduction band of ZnO.

The catalytic activity was increased by 71% through the addition of the catalyst dose from 0.25 to 0.5 g/L, due to proportional increase of available active sites. When the dose was increased to 1.0 g/L, there was a relative decrease in the rate of reaction because of light scattering by the highly turbid suspension and particles' shading effect. The impact of light intensity was found to be increasing with the decrease of quantum yield.

The rate of CO production (152.6 µmol g^{-1} h^{-1}) for the green synthesized ZnO/CuO nanocomposite of this study was considerably higher than those of similar published research works for ZnO-based materials. Moreover, the stable performance (91.8%) for five consecutive cycles is markedly better compared to those of the composite materials synthesized using the hydrothermal process. This improved activity is due to the creation of the S-scheme heterojunction using plant biomolecules, thereby improving the charge

Table 6. Effect of key operational parameters on the photocatalytic CO_2 reduction rate over ZnO/CuO nanocomposite.

Parameter Varied	Value	CO Evolution (µmol g^{-1} h^{-1})	CH_4 Evolution (µmol g^{-1} h^{-1})	CH_3OH Evolution (µmol g^{-1} h^{-1})
Catalyst Dosage	0.25 g/L	89.3	34.5	15.2
	0.50 g/L	152.6	61.8	34.4
	1.00 g/L	135.1	54.7	28.9
Light Intensity	50 mW/cm²	64.2	22.1	9.5
	100 mW/cm²	152.6	61.8	34.4
	150 mW/cm²	161.3	65.2	36.7
CO_2 Flow Rate	50 mL/min	152.6	61.8	34.4
	100 mL/min	141.7	56.4	30.1

Table 7. Comparative analysis of the ZnO/CuO nanocomposite performance in photocatalytic CO_2 reduction with recent state-of-the-art ZnO-based photocatalysts.

Photocatalyst & Synthesis Method	Light Source	Reaction Medium	Main Products & Max. Yield (µmol g^{-1} h^{-1})	Reusability (Cycles)	Reference & Year
ZnO/CuO (Biogenic S-Scheme)	300 W Xe, AM 1.5G	$H_2O/NaHCO_3$	CO (152.6), CH_4 (61.8), CH_3OH (34.4)	5 (91.8% retention)	This Work
ZnO/CuO Hydrothermal	300 W Xe	H_2O Vapor	CO (98.3), CH_4 (12.1)	4 (85% retention)	[10], 2023
ZnO/g-C_3N_4 Calcination	300 W Xe, Visible	H_2O/TEOA	CO (45.2), CH_4 (18.5)	3 (78% retention)	[16], 2022
ZnO/TiO_2 Electrospinning	350 W Xe	H_2O	CO (82.4), CH_4 (28.9)	5 (83% retention)	[9], 2024
ZnO/Ag NPs Photodeposition	300 W Xe	NaOH Solution	CH_3OH (18.7), CO (67.8)	3 (70% retention)	[20], 2021

transport and separation efficiency.

CONCLUSION

In this study, the successful synthesis of the ZnO/CuO nanocomposite with S-scheme configuration through two-stage green synthesis methodology involving the leaf extract of Rhus coriaria endemic to Uzbekistan and the assessment of its photocatalytic activity in CO_2 reduction under simulated sunlight irradiation were accomplished. As a result of XRD study, it could be stated that hexagonal ZnO phase with crystallite size of 24.1 nm and monoclinic CuO phase with crystallite size of 15.3 nm were formed simultaneously without any additional phases. EDS study showed that the surface Zn/Cu ratio was 1.89 and there was also 3.39 wt% organic carbon on the surface of the nanocomposite coming from plant-based compounds which is of utmost importance regarding colloidal stability and surface charge transfer which also has been found in [9]. The BET specific surface area of the nanocomposite was found to be 37.3 m²/g, which is 216% higher than pure ZnO (11.8 m²/g). The findings of DRS spectroscopy and Tauc plot measurements showed that there was a considerable reduction in energy gap from 3.19 eV of ZnO to 2.65 eV for the nanocomposite while the position of conduction band was found to be -0.38 eV with respect to NHE. It should be noted that this energy gap value could be considered as thermodynamically favorable for multi-electron reduction of CO_2 molecules. In addition, photoluminescence spectrum revealed which is consistent with the findings of [14] that considerable intensity reduction of the emission peak for the composite as compared to that of pure ZnO sample and this fact clearly indicates charge carriers recombination inhibition due to charge separation in the heterogeneous p-n junction. Performance evaluation of photocatalytic properties at optimum operating parameters (0.5 g/L catalyst concentration, 100 mW/cm2 light intensity, and 50 mL/min flow rate of CO_2) indicated that the ZnO/CuO nanocomposites exhibited carbon monoxide conversion rate of 152.6, methane conversion rate of 61.8, and methanol conversion rate of 34.4 μmol/g/h. This indicated that there was an increase by 8.2 times, 10 times, and 19.1 times when compared to pure ZnO catalyst. Selectivity towards methanol conversion increased from 6.7% to 13.8%. These findings are in line with [27]. To conclude, the green synthesis methodology was applied in this study. Which use abundant plant materials available in Uzbekistan, not only offers an inexpensive and environmentally friendly process to synthesize metal oxide nanocomposites, but also achieves a remarkable efficiency for converting CO_2 into solar fuels through accurate tuning of the heterogeneous S-scheme coupling. Indeed, the achieved rate of 152.6 μmol/g/h of CO production is a significant achievement relative to those reported recently in the literature. This is evident proof of great prospects that such a composite may offer for its practical use in industry. Future research can be aimed at optimizing the phase ratio, designing a continuous photocatalytic reactor, and studying the surface reactions' mechanisms in detail with the help of in situ spectroscopy methods.

CONFLICT OF INTEREST

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

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