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Performance optimization of CuO-ZnO ceramic electrode on the electrocoagulation of wastewater

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ABSTRACT

Copper oxide (CuO) and zinc oxide (ZnO) were used as a matrix and reinforcement in the compression molding technique to produce conductive ceramic electrodes. CuO-ZnO-based ceramic composites were fabricated through conventional pressing with CuO:ZnO ratios of 75 %:25 %, 80 %:20 %, 85 %:15 %, 90 %:10 %, and 95 %:5 %. CuO and ZnO powders were sieved, dried, mixed, and then compressed at 300 MPa for 10 min to form a pellet before being burned for 3 h at 800, 900, and 1000 °C in a furnace. The sample's density, porosity, and electrical conductivity were measured. In addition, SEM, XRD analysis, and palm oil wastewater electrocoagulation were carried out. The optimal density value of CuO-ZnO-based ceramic composites ranged from 8.564×10^3 to 9.205×10^3 kg m^{-3}. The optimal composition of the CuO:ZnO in the synthesized composites, namely 95 %:5 %, yielded the lowest porosity values (12.1 %–35.4 %), while the 75 %:25 % composition produced the highest porosity values (20.8 %–88.3 %). The optimum porosity value (12.1 %) was obtained by CuO:ZnO composite composition of 95 %:5 % with a sintering temperature of 1000 °C. This composite also generates a conductivity value of 1.019–16.897 S/m. The interaction of Zn^{2+} ions from ZnO crystals with CuO crystals increases the size of CuO and ZnO crystals, as determined by XRD analysis. Based on the findings of this study, CuO-ZnO ceramic electrodes could be utilized in industrial wastewater treatment as the water quality test result met the requirements of Minister of Environment Decree No. 51/MENLH/10/1995.

1. Introduction

Electrocoagulation is a complex technique that utilizes various processes to remove water contaminants [1]. Electrocoagulation provides an alternative to adding metal salt, polymer, or polyelectrolyte to destabilize emulsions and suspensions. Using highly charged polymeric metal hydroxide species, electrocoagulation has been utilized to remove metal, colloidal solids, particles, and soluble inorganic contaminants from wastewater [2]. Metals commonly used in the electrocoagulation process, such as Al, Cu, and Fe, have several drawbacks, including the fact that they cannot be used to treat wastewater with high electrolyte properties because a short circuit will occur between the electrodes and the anode rods, which are easily corroded and eroded (reduction) [3] to diminish the performance of the metal plate to the point where it must be continually replaced [4]. Therefore, this study attempted to fabricate a conductive ceramic electrode from CuO and ZnO composites. Composite material is a combination of two or more materials selected based on a combination of the physical properties of the constituent materials to produce a new material with superior physical, mechanical, and electrical properties to the basic material properties before mixing and surface bonding between each constituent material [5]. Ceramic Matrix Composite (CMC) is one of several comaterials frequently used based on the type of matrix [6]. CMC is an alternative material because its dimensions are stable and even more durable than those of metal, it is extremely tough [7], its surface characteristics are wear-resistant [8], its chemical elements are stable at high temperatures [9], and possesses high corrosion resistance [10].

Copper oxide (CuO) and zinc oxide (ZnO) were used as a matrix and reinforcement in the compression molding process in order to produce these conductive ceramic electrodes. Different calcination temperatures of ZnO-CuO nanocomposites may be initiated by the divergent and regulated ion distribution on the 'honeycomb' porous structure, according to a study by Govindasamy et al. [11]. CuO is an oxide of a transition metal. CuO is a p-type semiconductor material with a monoclinic structure and a narrow bandgap of

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1.2 eV (indirect) [12]. Due to its band gap energy of 1.4 eV, it is also desirable for light-harvesting applications [13]. Significant energy gaps can withstand large electric fields and high temperatures, which is why they are frequently utilized in the ceramics industry [14].

Meanwhile, ZnO is an eco-friendly material since it is compatible with living organisms and has a wide range of applications without harming human health or the environment [15]. In addition, it is highly effective at removing and completely degrading environmental pollutants. The ability of ZnO to absorb a vast portion of the solar spectrum and more light quanta than other metal-oxide semiconductors is its most significant advantage. ZnO is a typical semiconductor having a broad band gap of 3.37 eV, a high excitation binding energy of 60 meV at ambient temperature, a low price relative to other metal oxides, great photosensitivity, and environmental stability [16]. Due to its unique chemical and physical properties, ZnO provides a high driving power for reduction and oxidation reactions. Therefore, the conductive ceramic made from these two materials can be used as an alternative material for wastewater treatment.

As electrodes, the production of conductive ceramic electrodes based on CuO-ZnO composites that are distinguished by their physical properties, mechanical properties, electrical properties, microstructure, and crystal structure is anticipated [17] so that it can be utilized as an alternative electrode through electrocoagulation following the Minister of Environment Decree No. 51/MENLH/10/1995 in treating industrial wastewater [18].

2. Materials and methods

2.1. Materials

Materials used in this research were CuO powder (Pudak), ZnO (Merck), and palm oil wastewater. Meanwhile, the equipment used was a 100-mesh sieve, beaker glass, digital scale, spatula, furnace (High Temperature Furnace), sample mold, hydraulic press, Universal Testing Machine, PSA (Power Supply Adapter), digital multimeter, crocodile clamp, probe, chamber, XRD (X-ray Diffractometer), pH meter, TSS meter, AAS (Atomic Adsorption Spectrophotometer), water sample container (size: 5 × 10 × 10 cm), and electrode support.

2.2. Experimental

2.2.1. Manufacturing and characterization of CuO-ZnO as conductive ceramic electrodes

Copper oxide and ZnO powders were sieved using a 100-mesh screen. Those powders were then thoroughly combined for 5 min using a dry mixing method. CuO and ZnO mass ratios were 75 %:25 %, 80 %:20 %, 85 %:15 %, 90 %:10 %, and 95 %:5 %, respectively. Homogeneous materials were compressed in a hydraulic press at 300 MPa for 10 min to form a pellet. The pellet was burned for 3 h in a furnace at temperatures of 800, 900, and 1000 °C. The properties of the composites were then determined by measuring the sample's density, porosity, and electrical conductivity. Additionally, XRD was utilized for crystalline structure analysis.

2.2.2. Wastewater treatment process

The water quality of wastewater samples was evaluated, including color, presence of Cu metal, total suspended solids (TSS), pH, BOD, COD, oil, and fat. The sample waste was prepared in a glass container, and two parallel pairs of conductive ceramic electrode plates were arranged 1.5 cm apart in the bath. A voltage of 12 V was applied to a ceramic electrode by connecting it to a voltage source (PSA) to initiate electrocoagulation (see illustration in Fig. 1). After that, the sample was observed for 90 min, and then the parameter was tested and analyzed.

3. Results and discussion

3.1. SEM characterization

In order to observe the surface morphology of the electrodes, the CuO:ZnO composite microstructure was characterized. From the test results characterization of the physical, mechanical, and electrical properties of the CuO-ZnO conductive ceramic electrode material, the optimal composition is 95 %:5% CuO:ZnO sintered at 1000 °C. However, for the outcomes of these tests, microstructural testing is required to determine the optimum electrodes based on the characterization values so that these materials can be utilized as electrodes in industrial wastewater treatment. CuO: ZnO composites with composition variations of 75 %:25 %, 85 %:25 %, and 95 %:5% were sintered at 800, 900, and 1000 °C, respectively. The test's findings, which were conducted using SEM EVO MA 10 with an object magnification of 500 times, are depicted in Fig. 2.

The unfavorable results are depicted in Fig. 2b, which shows an image with pores so large that the material's physical, mechanical, and electrical properties are not suitable. Fig. 2d depicts the optimal SEM test result: the CuO:ZnO composite sample with a composition of 95 %:5% and a sintering temperature of 1000 °C, where the image has a denser surface structure and fewer pores than the other samples. Because the ZnO-reinforced CuO composite was spread equally during the production process, the results can alter the sample's physical and mechanical qualities as well as its electrical properties.

3.2. Density

Based on Fig. 3, the optimal density value occurs at a CuO-ZnO composition of 95 %:5 %, with density values ranging from 8.564×10^3 to 9.205×10^3 kg m^{-3}. Meanwhile, the theoretical densities of CuO and ZnO are 6.505×10^3 and 5.606×10^3 kg m^{-3}. The less optimal density value for CuO-ZnO composition is 75 %:25 %, with values between 7.280×10^3 and 7.872×10^3 kg m^{-3}. The density value obtained is influenced by the addition of ZnO reinforcing composition compared to the density value of CuO centrifuged at 1000 °C without any binder, which has a density value of 8.322×10^3 kg m^{-3}. This occurs because the ZnO reinforcing powder can combine with other compounds (flexible) [19]. This is due to the presence of cohesive bonds (adhesion-cohesion), as well as the interface between the ZnO reinforcing particle and the CuO matrix, electrostatic forces, and Van der Waals forces [20]. The surface roughness of the particles influences the interlocking between the reinforcement and matrix. In this instance, the cohesiveness between CuO powder and ZnO is responsible for the binding. The appropriate density was achieved because of the compaction pressure, which increased interlocking. Meanwhile, the electrostatic bond is formed due to the friction between the particle surfaces resulting from the compaction procedure (pressure). The magnitude of the applied stress affects van der Waals force. When the applied compressive force in the interfacial interaction between reinforcing particles and the matrix is less than the matrix's yield strength and the reinforcement's yield strength, the matrix and reinforcement deform elastically, resulting in a low-density value [21].

Temperature treatment during sintering also affects the density value. The sintering temperature demonstrates that the density value is directly proportional to the addition of the sintering temperature treatment, with the density increasing as the temperature is increased [22]. The observation results in high-density values at

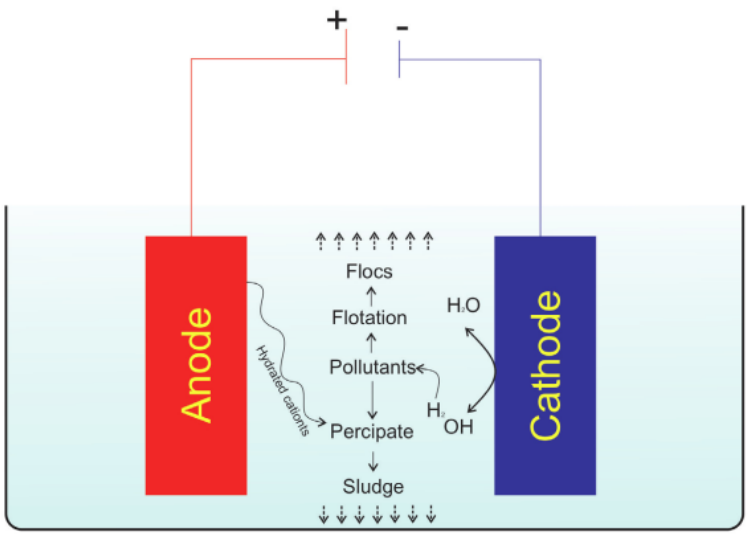

Fig. 1. Electrocoagulation process.

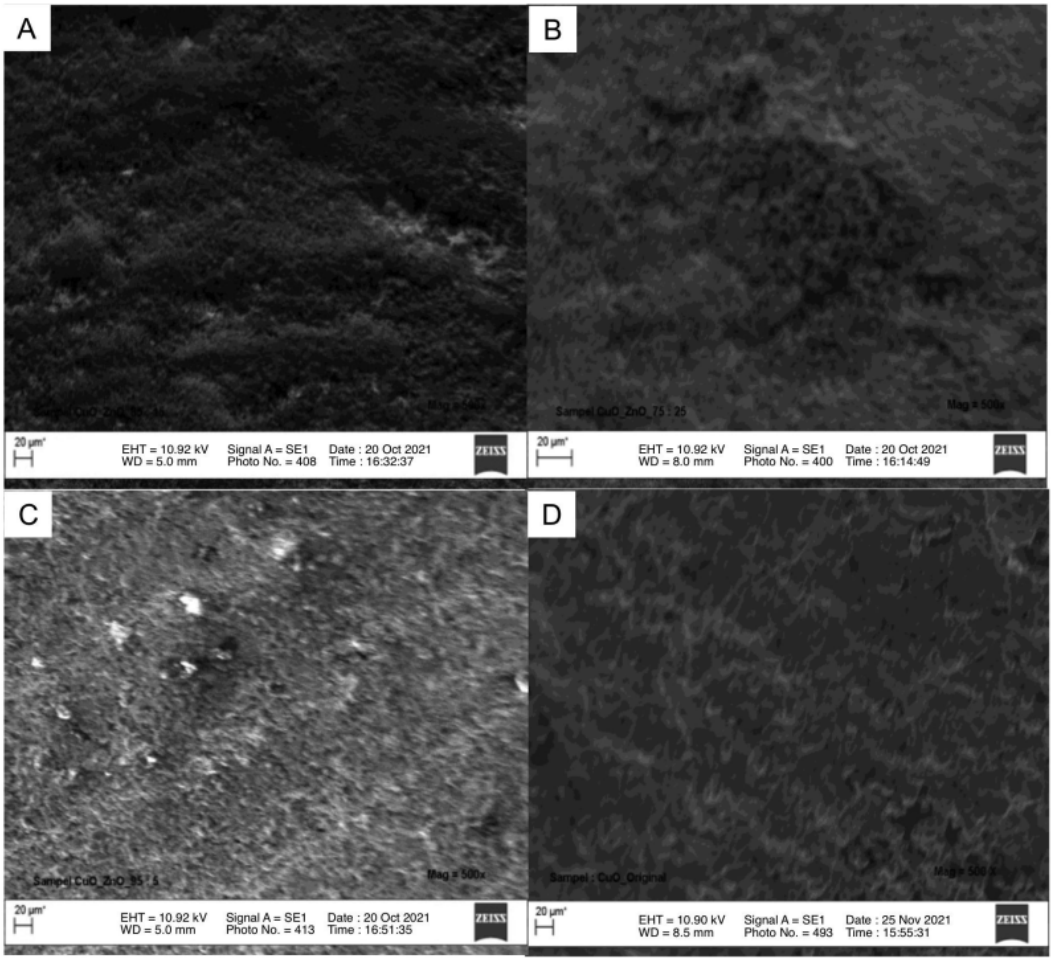

Fig. 2. SEM micrograph analysis of (A) CuO 100 % at sintering temperature of 1000 °C, (B) CuO:ZnO 75 %:25 % at sintering temperature of 800 °C, (C) CuO:ZnO 85 %:15 % at sintering temperature of 900 °C, and (D) CuO:ZnO 95 %:5 % at sintering temperature of 1000 °C.

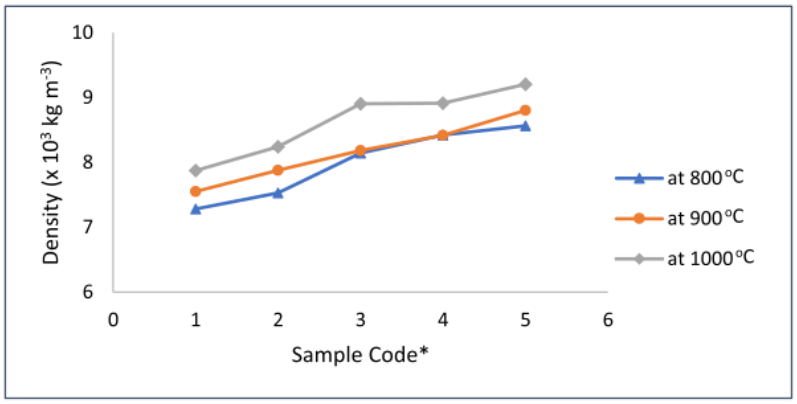

Fig. 3. Relationship between density and the composition of CuO:ZnO at various sintering temperatures (*1 = 75 %:25 %, 2 = 80 %:20 %, 3 = 85 %:15 %, 4 = 90 %:10 %, 5 = 95 %:5 %).

a combustion temperature of 1000 °C were obtained from 8.564 × 10^3 to 9.205 × 10^3 kg m^{-3}. The low-density value at 800 °C treatment goes from 7.280 × 10^3 to 7.872 × 10^3 kg m^{-3}. This occurs because, when exposed to a high temperature, the atoms that make up the composite material undergo diffusion, causing the atoms between the reinforcement and the matrix to form a strong bond and solidify, causing the pores to contract and become tight.

3.3. Porosity

The porosity is the volume of empty voids expressed as a percentage. Density is also linked to porosity [23]. In this study, the porosity test on the CuO-ZnO conductive ceramic electrode material is conducted in accordance with ASTM C 373–88. Table 1 presents the porosity values for each treatment.

Based on Table 1, the relationship between the porosity value of the CuO-ZnO conductive ceramic electrode material and the composition of CuO and ZnO at different sintering temperatures can be graphed, as shown in Fig. 4.

The optimum condition of the CuO-ZnO composite obtained a low porosity value, namely the CuO-ZnO composition of 95 %:5 %, with a porosity value ranging from 12.1 to 35.4 %. Meanwhile, the high porosity condition occurs in the CuO-ZnO composition of 75 %:25 %, with porosity values ranging from 20 to 88.3 %. It can be observed that the porosity value tends to decrease if the sintering temperature is increased, both with and without the addition of ZnO filler. During the combustion process, the pores shrink, and atomic bonds form, increasing the density value of the material and an increase in the porosity value of the material decrease [24]. Similarly, if the amount of ZnO filler is decreased to 5 %, the porosity value will decrease.

At a sintering temperature of 1000 °C, the range of porosity values for adding 5 % to 25 % ZnO filler is between 20 % and 12.1 %. Based on the best porosity values, the optimal CuO:ZnO composite composition was at 95 %:5% with a sintering temperature of 1000 °C, which generates a porosity value of 12.1 %.

3.4. Electrical conductivity

Electrical conductivity is a measurement of a material's ability to conduct electric current; its value is affected by the density of the atomic structure of the material that composes it, the number of electron charges, and the level of purity [25]. The result of electrical conductivity is shown in Table 2. In addition, Fig. 5 demonstrates that optimal conditions resulted in high electrical conductivity values at a composition ratio of 95 %:5% with conductivity values ranging from 1.019 to 16.898 S/m. In comparison, less optimal conditions were obtained at 75 %:25 %, with conductivity values ranging from 0.69 to 2.24 S/m. Furthermore, the conductiv-

Table 1
Porosity data for CuO-ZnO ceramic electrodes.

Temperature (°C)	Composition of CuO:ZnO (% mass)	m_d (×10^{-3} kg)	m_w (×10^{-3} kg)	Volume (×10^{-6} m^3)	Porosity (%)
800	75:25	2.366	2.653	0.325	88.3
	80:20	2.402	2.600	0.319	62.2
	85:15	2.304	2.475	0.283	60.5
	90:10	2.350	2.474	0.279	44.4
	95:5	2.492	2.595	0.291	35.4
900	75:25	2.552	2.641	0.338	24.6
	80:20	2.308	2.380	0.293	24.2
	85:15	2.422	2.490	0.296	23.0
	90:10	2.449	2.513	0.291	22.0
	95:5	2.553	2.616	0.290	21.7
1000	75:25	2.283	2.341	0.290	20.0
	80:20	2.373	2.429	0.288	19.4
	85:15	2.529	2.577	0.284	16.9
	90:10	2.727	2.768	0.306	13.4
	95:5	2.743	2.779	0.298	12.1

Note: m_d (dry mass), m_w (wet mass).

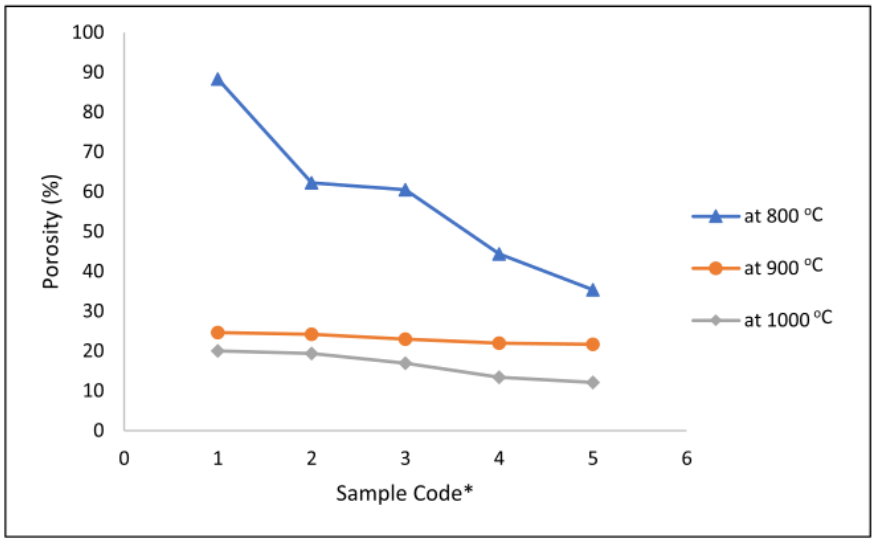

Fig. 4. Relationship between porosity and the composition of CuO:ZnO at various sintering temperatures (*1 = 75 %:25 %, 2 = 80 %:20 %, 3 = 85 %:15 %, 4 = 90 %:10 %, 5 = 95 %:5 %).

Table 2
Electrical conductivity measurements of CuO-ZnO conductive ceramic electrodes.

Temperature (°C)	Composition of CuO:ZnO (%)	σ (S/m)
800	75:25	0.694
	80:20	0.769
	85:15	0.801
	90:10	0.815
	95:5	1.019
900	75:25	1.113
	80:20	1.563
	85:15	1.683
	90:10	1.842
	95:5	2.175
1000	75:25	2.245
	80:20	2.860
	85:15	2.924
	90:10	3.561
	95:5	16.898

ity range for the 1000 °C sintering temperature treatment is between 2.245 and 16.989 S/m, whereas the conductivity range for the 800 °C combustion temperature treatment is between 0.69 and 1.01 S/m. In addition, CuO sintered at 1000 °C without any binder has a conductivity value of 4.58 S/m.

The value of electrical conductivity increases with increasing temperature, as shown in Fig. 5. Likewise, based on Fig. 5, electrical conductivity also increases along with the decrease of ZnO filler. Although, it did not affect the mechanical properties. This indicates that the ZnO powder is insoluble and will degrade at the grain boundary. This degraded ZnO powder will inhibit the growth of CuO matrix grains during the temperature treatment [26]. As a result, the resulting composite will have a more refined grain structure, which will decrease the average free path of the charge carrier and result in a high resistivity (small electrical conductivity) [27]. Moreover, the inhomogeneity of the material's composition results in impurities that cause the material to be riddled with holes, thereby preventing the movement of electrons [28].

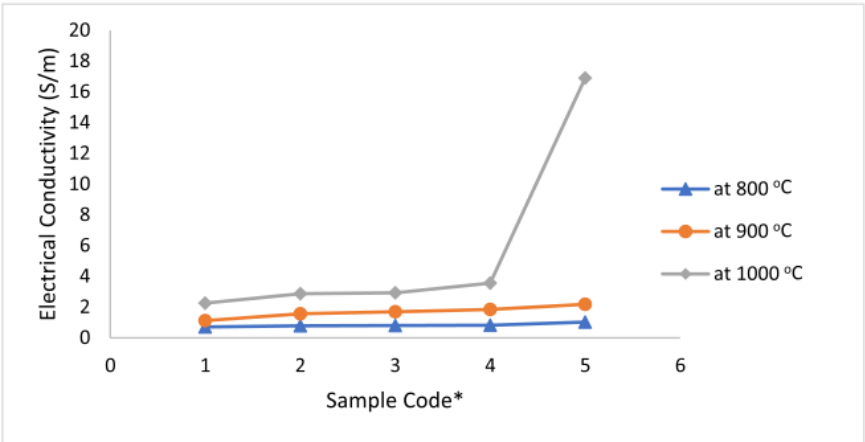

Fig. 5. Graph of the relationship between electrical conductivity and composition variations of several sintering temperature treatments (*1 = 75 %:25 %, 2 = 80 %:20 %, 3 = 85 %:15 %, 4 = 90 %:10 %, 5 = 95 %:5 %).

3.5. Particle size of the material

Our previous work has reported the XRD characterizations of the CuO-ZnO composites with various compositions and sintering temperatures [29]. This section further analyses the fabricated materials. The *Match 1.1.1* software was utilized to analyze the three compositions based on the highest peak at the diffractogram. *Match* software analysis of CuO-ZnO composites at compositions of 75 %:25 % (800 °C), 85 %:15 % (900 °C), and 95 %:5% (1000 °C) reveal the presence of CuO in the composite by modifying the JCPDS standard No. 80–0076. CuO has lattice parameters of a = 4.679; b = 3.431, c = 5.136, and a monoclinic crystal. By adjusting the JCPDS standard No. 05–0664, the ZnO in the composite has lattice parameters of a = b = 3.25 and c = 5.216, and a hexagonal crystal structure.

The particle size was derived from XRD diffraction pattern by calculating the diffraction peaks' FWHM (Full Width at Half Maximum) using the Scherrer equation [30]. The results of the calculation of the crystal size (D) of CuO and ZnO of the CuO-ZnO composite with a composition of 75 %:25 % (800 °C), 85 %:15 % (900 °C), and 95 %:5 % (1000 °C) at the highest peak of the diffraction pattern are presented in Table 3.

Table 3 shows that the size of CuO and ZnO crystals increase due to the interaction of Zn^{2+} ions from ZnO with CuO crystals. The larger the size of the crystal, the quicker the charge-carrying process (the greater the electrical conductivity) and the maximum capacity to conduct electric current [31].

3.6. Wastewater electrocoagulation process with conductive ceramic electrodes

This study used conductive ceramic electrodes based on CuO-ZnO composites to electrocoagulation wastewater from the palm oil industry. These composites had the ideal composition and sintering temperature at a ratio of 95 %:5% CuO:ZnO and at a sintering temperature of 1000 °C. The electrodes used in the electrocoagulation process of wastewater treatment have been characterized and have the best physical, mechanical, electrical, microstructure, and crystal structure properties. The wastewater used in this study is the palm oil industry wastewater. The blackish-brown wastewater is tested for several parameters before electrocoagulation treatment to determine whether it is suitable for use in accordance with wastewater and quality standards. The electrocoagulation method was used to treat the wastewater after it had been tested. The electrocoagulation process in wastewater treatment was carried out in an aquarium with a size of 5 × 10 × 10 cm. The electrocoagulation procedure took 90 min to complete. Susilawati et al. [29] also used copper (Cu) metal plate electrodes in the electrocoagulation process to treat peat water for 45 min electrocoagulation process. This time is significantly more effective, but at this point, the Cu metal plate dissolves in water at a rate that exceeds the standard. In addition, Cu metal is frequently corroded and must be constantly replaced as the performance of the electrode degrades with repeated use. Besides, Chavalparit and Ongwandee [32] researched electrocoagulation for the treatment of biodiesel waste. They concluded that the electrocoagulation process could decrease COD by 2.43 %, oil and fat by 98.4 %, and suspended solids by 96.59 %, with a pH of 6.06. With an applied voltage of 18.2 V, the required electrocoagulation process time was 23.5 min. Additionally, according to Augustin [33], electrocoagulation based aluminum electrodes and NaCl were favorable to reduce turbidity, acidity, BOD, COD, and heavy metals from palm oil mill waste at the Chumporn Province in Thailand.

Compared to Cu and aluminum metal plates, the conductive ceramic electrodes based on CuO-ZnO composites required much more time. However, this electrode is resistant to corrosion and robust for numerous applications. Although we have not investigated the corrosion resistance of this material, ceramic electrodes are theoretically more resistant to corrosion than metal electrodes. Regarding this, Table 4 provides pH parameter testing performed five times using CuO-ZnO conductive ceramic electrodes.

Table 4 shows that the pH value of conductive ceramic electrodes based on CuO-ZnO composites after five times usage in the electrocoagulation process was relatively constant. This may indicate that conductive ceramic electrodes have durable properties similar to the fundamental properties of ceramics. In addition, several parameters, including color, TSS, pH, BOD, COD, oil and fat, and the amount of dissolved Cu in the water, were obtained to reassess the quality of the processed palm oil wastewater following electrocoagulation. The test results, as presented in Table 5, are compared to the wastewater standard suggested by the Minister of Environment Decree No. 51/MENLH/10/1995.

Table 5 indicates that the color parameter decreased by 89.29 % from 1967.5 to 210.6 TCU. The content of dissolved Cu metal in wastewater increased from 0.05424 to 0.07188 mg L^{-1}, but the increase was slight. In addition, TSS decreased from 570 to 164 mg L^{-1} by 71.22 %; pH increased from 4.3 to 7.4; BOD decreased from 595 to 74 mg L^{-1} by 87.56 %; COD decreased from

Table 4
Measurement results of pH parameters several times using conductive ceramic electrodes based on CuO-ZnO composites in the electrocoagulation of wastewater.

Repetition*	Resulted pH
1	7.4
2	7.4
3	7.38
4	7.45
5	7.5

*Processing time for all experiments was 90 min.

Table 3
Diameter size of CuO and ZnO crystals in CuO-ZnO composite.

CuO-ZnO composite	Peak	FWHM (Deg)	CuO			ZnO		
			θ (Deg)	D (nm)	D_{avg} (nm)	θ (Deg)	D (nm)	D_{avg} (nm)
75 %:25 % at 800 °C	1	0.00279	17.72	41.414	40.8864	15.82	40.9971	41.2583
	2		19.31	41.805		18.06	41.2847	
	3		24.31	39.438		28.33	41.4932	
85 %:15 % at 900 °C	1	0.00348	17.79	51.635	52.5141	15.885	51.1255	52.914
	2		19.40	52.116		17.22	51.7332	
	3		24.32	53.969		18.13	55.8831	
95 %:5% at 1000 °C	1	0.00279	17.80	51.657	52.6237	17.37	51.5380	53.709
	2		19.44	52.161		23.64	53.6930	
	3		24.50	54.052		28.36	55.8958	

Table 5
Characteristics of palm oil wastewater before and after treatment by electrocoagulation method using based on CuO-ZnO composites-based conductive ceramic electrodes.

Parameter	Test result				Standard palm wastewater
	Unit	Before	After	% Decrease	
Colour		1967.5	210.6	89.29	–
Cu	mg L^{-1}	0.05424	0.07188	–	–
Total suspended solids (TSS)	mg L^{-1}	570	164	71.22	250
pH	–	4.30	7.4	–	6.0–9.0
BOD	mg L^{-1}	595	74	87.56	100
COD	mg L^{-1}	1840	236	87.173	350
Oil and fat	mg L^{-1}	33	15	54.54	25

1840 to 236 mg L^{-1} by 87.173 %; and oil and fat decreased from 33 to 15 mg L^{-1} by 54.54 %. According to the obtained data, the wastewater treated by the electrocoagulation process using CuO-ZnO composites-based conductive ceramic electrodes has met the standard issued by the Minister of Environment Decree No. 51/MENLH/10/1995.

4. Conclusion

In this study, the optimal density value of CuO-ZnO-based ceramic composites was obtained with a CuO:ZnO composition of 95 %:5% ranging from 8.564 × 10^3 to 9.205 × 10^3 kg m^{-3}. On top of that, temperatures also influence the density value, with composite sintered at 1000 °C possessing higher density than those at 800 and 900 °C. The optimum composition of the CuO-ZnO composite (95 %:5%) results in a low porosity value, with a porosity value ranging from 12.1 to 35.4 %. In comparison, high porosity was obtained from the 75 %:25 % CuO-ZnO composition, with porosity values ranging from 20 to 88.3 %. The optimal CuO:ZnO composite composition was 95 %:5%, based on the best porosity values (12.1 %) with a sintering temperature at 1000 °C. This composition also produces CuO-ZnO with high conductivity values ranging from 1.019 to 16.898 S/m. Based on the XRD analysis of the composites, it is concluded that the interaction of Zn^{2+} ions from ZnO with CuO crystals increases the size of CuO and ZnO crystals. In addition, it can be concluded that conductive ceramic electrodes based on CuO-ZnO composites can be used in industrial wastewater treatment through the electrocoagulation method. Based on the water quality testing results, the data indicate that the treated wastewater has met the standard issued by the Minister of Environment Decree No. 51/MENLH/10/1995.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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