

# **Chemical Engineering & Process Techniques**

### **Research Article**

# Simultaneous Reduction of CO<sub>2</sub> and Brilliant Green Dye Removal Electrochemically in Bicarbonates of Sodium and Potassium Salts

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### **Abstract**

A new approach of experimental studies for simultaneous  $\rm CO_2$  reduction and Brilliant Green (BG) dye removal electrochemically was studied on Zinc (Zn) and Tin (Sn) cathodes and a common Cobalt oxide ( $\rm Co_3O_4$ ) anode in 0.5M (10 ppm BG) KHCO $_3$ /NaHCO $_3$  electrolytes. Formic acid was the only product formed along with removal of BG dye was observed at different applied voltages. High Faradaic efficiencies were observed at low applied voltages with low BG dye removal. Conversely, high voltages were favorable for maximum dye removal with low HCOOH acid Faradaic efficiencies. For Sn as cathode maximum Faradaic efficiencies of HCOOH in NaHCO $_3$  is obtained to be 76.3% (5 min) – 2 V, BG dye removal using Sn as electrocatalyst in NaHCO $_3$  electrolytes was obtained to be 94.9% (25 min) – 3.8 V. The present studies demonstrate the most effective process for conversion of atmospheric  $\rm CO_2$  to HCOOH and the remedy for purification of impure water.

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# Keywords

- CO, reduction
- Faradaic efficiency
- Brilliant Green (BG) dye
- Tin (Sn)
- Zinc (Zn)
- Formic acid (HCOOH)

# INTRODUCTION

Global warming effect was caused by the combustion of fossil fuels, which releases greenhouse gases during energy generation which causes great damage to the society. The different gases like methane, nitrous oxide, other halocarbons and CO<sub>2</sub> were evolved during the process. Of which CO<sub>2</sub> is the main contributor to the global warming effect [1-3]. There is a need to reduce these CO2 before it causes damage to the environment [4,5]. Several methods were in existence towards reduction of CO<sub>2</sub> to different products, the main focus went on electrochemical CO<sub>2</sub> reduction because of its several advantages [6,7]. The studies reported that reduction depends on different conditions like electrocatalyst, electrolytes, and applied voltages [8-10]. Irabein et al., studied the work on different copper based electrocatalysts for liquid product methanol generation with high efficiencies [35,36]. Copper is accepted as a best electrocatalyst in reducing CO<sub>2</sub> to hydrocarbons, but due to multiple product formation the system becomes complex [11,12]. Some studies were done using Zn, Sn and Pb selectively form HCOOH as main product reduces the system complexity [13-17]. Lu et al., given a review in which discussed a reaction mechanism with effective experimental conditions on electrocatalyst type, reactor design and applied conditions for reduction of CO,

towards HCOOH formation electrochemically [18]. The effect of CO2 reduction to HCOOH was studied on synthesized lead oxide electrocatalyst at different applied voltages in KHCO3 and NaHCO<sub>3</sub> electrolytes. Guerra et al., studied the continuous reduction process for HCOOH formation using a lead cathode in ambient conditions. It was reported that electrolyte type used for reaction medium may also improve the process. Mainly, ionic liquids, place a major role in future investigations as it has a high CO<sub>2</sub> solubility and best electrochemical properties [20,37] Wang et al., studied the CO<sub>2</sub> reduction on Sn electrocatalyst for HCOOH formation in microbial cell. It was reported that Sn loaded gas diffusion electrodes were favorable towards the process with low energy consumption with purification of water [21]. Synthesis of Sn and Zn electrocatalysts by electrodeposition method and their applications for electrochemical reduction of CO2 to HCOOH in sodium and potassium based salts at different applied voltages were reported [22,23]. Zhang et al., studied the effective CO<sub>2</sub> reduction on different types of nano structured Sn electrocatalysts for HCOOH formation. High Faradaic efficiency for HCOOH formation from reduction of CO<sub>2</sub> at low over potential on Pd-Pt electro catalyst particles were reported [24]. Peng et al., studied the simultaneous CO2 reduction and the methyl orange dye removal using nitrogen doped tin oxide, Pt and copper as



electro catalysts [25,26]. The removal of BG dye removal along with  ${\rm CO_2}$  reduction is extremely significant because dye water from textile industries can be used as a reactant for  ${\rm H^+}$  generation instead of using pure water for oxidation reaction. This gives better solution for purification of water by removing the dye from the waste water by oxidation reaction at anode. Platinum was widely accepted as anode catalyst for water oxidation reaction to generate protons, however, in the present studies was done with  ${\rm Co_3O_4}$  as an anode which is cheap alternate electro catalyst [27-30].

From the literature studies, it was clearly confirmed that bicarbonate based solutions were favorable for  $\mathrm{CO}_2$  reduction due to high solubility of  $\mathrm{CO}_2$  [14-17]. The present study reports for the first time on simultaneous  $\mathrm{CO}_2$  reduction and BG dye removal in bicarbonate solutions using Sn and Zn as cathode with  $\mathrm{Co}_3\mathrm{O}_4$  anode. The effect of Zn and Sn electrocatalyst were studied separately for reduction and dye removal were studied with respect to time and different applied voltages. All studies were done using a 2-electrode glass cell and respective optimized results were reported clearly. The studies done here may be useful to initiate the process for  $\mathrm{CO}_2$  reduction to HCOOH with water purification using the selected catalyst combination for future applications.

# **MATERIALS AND METHODS**

## **Materials**

Graphite plates (1.5 x 2.5) cm $^2$  as a base for catalyst from Sunrise Enterprises, Mumbai. NaHCO $_3$ , KHCO $_3$ , iso-propyl alcohol and Brilliant green dye - Merck, India, Nafion (5wt %) - DuPont, USA. Chemicals (without any purification) and deionized water were used here for all experimental studies.

# Preparation of catalyst coated graphite electrodes for cathode and anode

The electrocatalysts were synthesized using electrodeposition method by applying a constant current between the two electrodes which were reported in our earlier studies [22,23]. The catalyst ink was prepared using synthesized electrocatalysts and coated on the graphite plates to prepare cathode and anode electrodes [22]. Adding 7.5 mg of the electrocatalysts in a binder solution [1:5 (nafion + IPA (Iso propyl alcohol) of 200  $\mu$ l] and sonicated for 30 min to get catalyst ink. Further the prepared ink was coated on graphite plate surface at the 80°C (2hr) at 2 mg/cm² catalyst loading to form an electrode. The same procedure was followed for all different catalyst loading.

# Electrochemical studies on electrochemical ${\rm CO_2}$ reduction and BG dye removal

A 2-electrode glass cell was used to study the electrochemical  $\mathrm{CO_2}$  reduction and BG dye removal. The setup used for the studies was shown in (Figure 1). 80 ml [0.5 M (10 ppm MB)] solution was bubbled with  $\mathrm{CO_2}$  for 50 min to get completely saturated  $\mathrm{CO_2}$  electrolyte solution and used the solution for the whole reaction without further bubbling. The anode and cathode were dipped in an electrolyte solution which was connected to a DC source for energy source. The studies were done at different applied voltages of 2, 2.3, 2.5, 2.8, 3.3 and 3.8 V with each voltage

at reaction times of 0-5, 10, 15, 20 and 25 min [19]. Faradaic efficiency was measured by using charge utilize for a product (HCOOH) to the total charge for the whole reaction.

FE= nFZ/Q, where n is moles of HCOOH formed, F-faradaic constant, Z is number of electrons involved in HCOOH formation reaction, Q-total charge of reaction

# Analysis of product and BG dye analysis

Ultra-fast liquid chromatography (UFLC) - [Shimadzu LC-20AD, UV-detector of deuterium lamp (SPD-20A)] is used at 205 nm wavelength for HCOOH analysis by using C-18 column (10X4 mm). 5mM Tetrabutyl ammonium hydrogen sulfate is used as mobile phase at 1 ml/min flow rate. MB dye removal analysis [UV-Visible Spectrophotometer (Perkin Elmer, Model: Lambda 35)].

# **RESULTS AND DISCUSSION**

# ${\rm CO_2}$ reduction and BG dye removal on Sn (cathode) and ${\rm Co_3O_4}$ (anode) electrocatalysts

The reduction of  ${\rm CO}_2$  and BG dye removal was studied using the Sn (cathode) and  ${\rm Co}_3{\rm O}_4$  (anode) electrocatalysts in bicarbonate based electrolytes. Maximum dye removal was observed along with higher Faradaic efficiencies for different applied voltages and the results were shown clearly. For all reaction studies, 0.5 M (10 ppm BG) concentration was used as electrolyte solution to conduct reaction.

Current density on applied voltage using Sn cathode: Bases on the applied voltages, current densities were shown which corresponds to the rate of reaction. The results in different electrolyte solutions were shown in (Figure 2A). Current densities were observed to be increasing with increasing voltages. That shows that the reaction rate is more at higher applied voltages which may be due to  $\mathrm{CO}_2$  reduction or hydrogen evolution. The effect of current density with different voltages shows the ability of selected electrocatalysts towards BG dye removal  $\mathrm{CO}_2$  reduction.

CO<sub>2</sub> reduction and BG dye removal in KHCO<sub>2</sub> solution: The experimental results in KHCO2 solution was shown in (Figure 2B,2D). For all applied voltages HCOOH was obtained as product with maximum dye removal. The reaction at 2V for reaction time of 5,10,15,20 and 25 min shows high efficiencies of 40.6, 29.4, 22.1, 36.6 and 22.8 % were obtained with dye removal (94.1, 94.6, 94.3, 94.3 and 94.2 %) were respectively. The variation in product concentrations with respect to time may due to the oxidation of HCOOH at anode [17]. At 2.3 V, Faradaic efficiencies (7.2, 14.7, 40.4, 6.64 and 8.6 %) and BG dye removal (93, 93.1, 93.1 and 93.44 %) were observed with the optimized condition for HCOOH formation is 40.4 % for reaction of 15 min. Lv et al., studied on HCOOH formation from reduction of CO2 on Sn electrode in KHCO<sub>2</sub> solution without any dye using Pt anode [17]. For reaction at 2.5 V, the decrease in efficiencies may be due to the evolution of hydrogen at cathode due to excess proton generation at anode. At 2.8 V reactions, though high removal was observed, but efficiencies were reduced with the optimized condition of 10.6 % for 5 min reaction. Koleli et al., studied the reduction studies electrochemically on Sn electrocatalyst using potassium

based electrolytes and reported the HCOOH at different applied voltages without any dye solution [14]. Very low efficiencies were observed at higher voltages (3.3 and 3.8 V) with high BG dye removal. The high applied voltages were not favorable for  $\mathrm{CO}_2$  reduction may be due to low  $\mathrm{CO}_2$  molecule concentration than hydrogen ions [23]. High efficiencies were observed at 2V which is the most optimized condition for simultaneous  $\mathrm{CO}_2$  reduction effective dye removal.

CO<sub>2</sub> reduction and BG dye removal in NaHCO<sub>3</sub> solution: Figure (2C,2E) shows the most effective results in sodium based electrolyte solution. It was observed that high Faradaic efficiencies were obtained for HCOOH formation compared with Potassium based solution with maximum dye removal. This is due to the higher solubility of CO<sub>2</sub> in this solution that participates in reduction reaction at the cathode surface than hydrogen evolution due to proton [15]. Faradaic efficiencies (76.3, 30.6, 36.3, 27.7 and 20.1 %) with BG removal of 92.8, 92.8, 93, 93.5 and 92.4 % were obtained at 2 V reactions. The optimized reaction for the maximum HCOOH formation is 76.3 % for reaction time 5 min. Chen et al., studied the effect of CO<sub>2</sub> reduction on Sn based electrocatalyst in CO2 saturated NaHCO3 solution for HCOOH generation [15]. At 2.3 V, the variation of Faradaic efficiencies may be due to formed product oxidation at Co<sub>3</sub>O<sub>4</sub> which leads to hydrogen evolution at the cathode [22,31]. At 3.3 V, low Faradaic efficiencies of 0.78, 1.05, 1.3, 1.1 and 1.5 % (Figure 2C) with simultaneous BG removal 93.3, 93.6, 94.06, 94.03 and 93.7 % were obtained. Zhang et al., studied the reduction process for HCOOH formation on nano Sn electrocatalysts using sodium based electrolyte [32]. The low efficiencies for HCOOH may be due to high hydrogen evolution at Sn electrode [19]. Faradaic efficiency (1.89, 1.8, 0.9, 0.46 and 0.39 %), BG removal (94.1, 94.06, 94.7, 94.8 and 94.9 %) were shown for reaction at 3.8 V. The optimized condition was 5 min reaction at which maximum efficiency of 1.89 % was obtained. The efficiencies were high in sodium based solution with effective reduction in low applied voltages and dye removal.

# ${\rm CO_2}$ reduction and BG dye removal on Zn (cathode) and ${\rm Co_3O_4}$ (anode) electrocatalysts

The simultaneous  $\mathrm{CO}_2$  reduction with BG dye removal was studied using Zn electrocatalyst as cathode. The studies were done in bicarbonate electrolyte solutions at different applied voltages to study the effective, optimized conditions towards maximum  $\mathrm{CO}_2$  reduction along with dye removal.

Current density on applied voltage using Zn cathode: The results for current density with respect to applied voltages for HCOOH Faradaic efficiencies in both electrolyte solutions were shown in (Figure 3). In particular the Current densities were increasing with applied voltage increase as shown in (Figure 3A). Electrolyte concentration of 0.5 M (10 ppm CV) was used to conduct electrochemical studies for all applied conditions. The current densities represent high reaction rates with maximum in NaHCO $_3$  electrolyte solution than KHCO $_3$ .

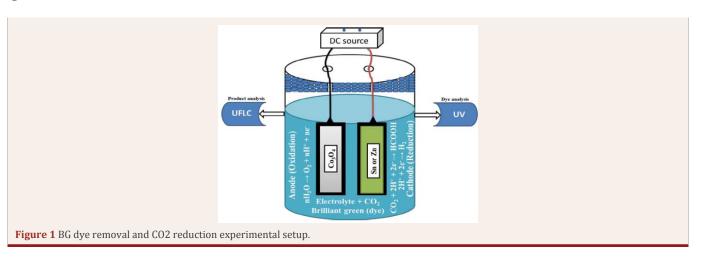
 ${
m CO}_2$  reduction and BG dye removal in KHCO $_3$  solution: The reaction in potassium based solutions was shown in (Figure 3B,3D). Hori et al., studied the reduction of  ${
m CO}_2$  for HCOOH formation on Zn electrocatalyst in KHCO $_3$  solution using Pt anode

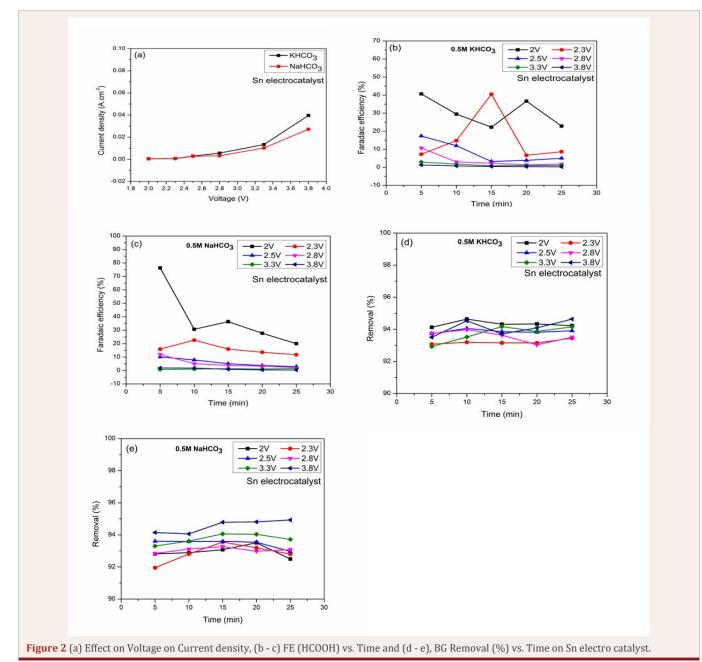
[31]. In all applied voltages, HCOOH was observed as an only product with maximum BG removal at the different reactions of 5, 10, 15, 20 and 25 min. At 2 V, maximum efficiencies were obtained at this voltage which is a clear sign for the best reaction condition. The change in Faradaic efficiencies with time in (Figure 3B) may be due to oxidation of the product (HCOOH) at a anode [17,22]. For reaction at 2.3 V, The optimized condition at this voltage is 15.01 % for 5 min reaction. Faradaic efficiencies [(10.8, 7.1, 3.2, 3.5 and 4.7 %), (5.1, 3.8, 3.7, 2.3 and 1.7)] were obtained at applied voltages of 2.5 and 2.8 V with BG removal [(92.1, 93.3, 93, 93.3 and 93.4 %), (92.9, 92.9, 93.8, 93.5 and 93.7 %)] respectively. Low efficiencies were observed at these voltages may be due to high hydrogen evolution at Zn electrode [33]. At 3.3, 3.8 V, very low efficiencies were seen compared with low applied voltages which may not be a better reaction condition towards CO<sub>2</sub> reduction in HCOOH formation. Electrochemical studies for HCOOH as product in CO<sub>2</sub> reduction on Zn electrocatalyst without dye in potassium salts were reported [22]. From the results it may be concluded that high applied voltages were not favorable for maximum HCOOH efficiencies. The result proves the capability of electrocatalyst towards CO2 reduction to HCOOH and maximum dye removal.

CO<sub>2</sub> reduction and BG dye removal in NaHCO<sub>3</sub> solution: The electrochemical studied results in NaHCO3 electrolyte solution were shown in (Figure 2C,2E). High efficiencies were observed at low applied voltages compared with higher voltages. Faradaic efficiencies (50.7, 26.9, 32.1, 19.3 and 28.1 %), BG dye removal (93.5, 93.3, 93.5, 93.5 and 93.5 %) were obtained at reaction time of 2 V. The optimized condition towards the maximum HCOOH formation is 50.7 % for reaction time of 5 min. The results show that with reaction times, efficiencies were changing may be due to HCOOH oxidation at anode [17,19]. Jin et al., studied the effect of CO<sub>2</sub> reduction on Zn electrocatalyst for HCOOH as product was reported [34]. At 2.3 V, low efficiencies were observed when compared with 2 V which may be due to high proton generation at Co<sub>2</sub>O<sub>4</sub> anode which converts into hydrogen at the cathode [31]. The reactions at 2.5 V, the optimized reaction condition are 17.4 % after a reaction time of 5 min for maximum HCOOH efficiency. HCOOH as product from CO2 reduction has been reported in KHCO<sub>2</sub> solution without dye on Zn electrocatalyst at different applied voltages [22]. Low Faradaic efficiencies were obtained at a applied voltage of 2.8 V (Figure 2D). The decrease in efficiencies may be due to low  ${\rm CO_2}$  reactant concentration molecules at cathode compared with protons [31]. Very low Faradaic efficiencies were obtained in 3.3 and 3.8 V may be due to high hydrogen evolution at the cathode surface [31]. The experimental results for CO2 reduction and BG dye removal for conditions were discussed and optimized conditions for HCOOH Faradaic efficiencies and BG dye removal for different reaction times with applied voltages were shown in (Table 1,2). For all the cases, low voltages were shown high Faradaic efficiencies for HCCOH formation.

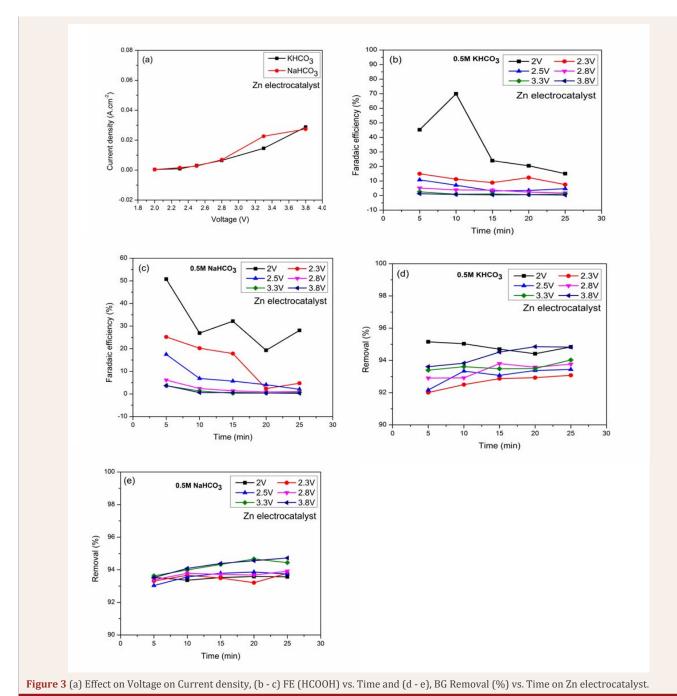
# **CONCLUSION**

A novel method for maximum BG dye removal and HCOOH Faradaic efficiencies were shown on Zn and Sn electrocatalysts with  ${\rm Co_3O_4}$  (anode). In both KHCO $_3$  and NaHCO $_3$  solutions HCOOH was formed as the only product for  ${\rm CO_2}$  reduction. Irrespective









**Table 1:** Maximum Faradaic efficiencies for HCOOH at different applied conditions.
 Maximum Faradaic efficiency (time) Voltage Sn Zn KHCO, NaHCO, NaHCO, KHCO<sub>3</sub> (V) (min) (%) (%) (min) (%) (%) (min) (min) 2 40.6 5 76.3 5 69.9 10 50.7 5 10 5 5 2.3 40.4 15 22.5 15 25.2 2.5 11.9 10 10.8 5 5 10.1 5 17.4 10.6 5 5 5 2.8 11.9 5 5.1 6.1 3.3 5 1.5 25 2.5 5 5 2.8 3.6 5 3.8 1.2 5 1.89 5 1.2 3.7 5



**Table 2:** Maximum BG dye removal at different applied conditions.

Voltage	BG removal (time)							
	Sn				Zn			
	KHCO <sub>3</sub>		NaHCO <sub>3</sub>		KHCO <sub>3</sub>		NaHCO <sub>3</sub>	
(V)	(%)	(min)	(%)	(min)	(%)	(min)	(%)	(min)
2	94.6	15	93.5	20	95.1	5	93.58	20
2.3	93.4	25	93.5	15	93	25	93.7	25
2.5	94	10	93.6	5	93.4	25	93.8	20
2.8	93.9	10	93.2	20	93.8	15	93.9	25
3.3	94.17	15	94.06	15	94	25	94.6	20
3.8	94.6	25	94.9	25	94.85	20	94.7	25

of applied voltages maximum BG dye removal was observed in all conditions for both (Zn and Sn) as electrocatalysts. HCOOH formation is favorable at low applied voltages compared with higher voltages. Maximum Faradaic efficiency of 76.3 % (5min) –2 V, BG dye removal 94.9 % (25 min) – 3.8 V for Sn electrocatalysts in NaHCO $_3$  electrolyte solutions. For Zn as electrocatalyst, maximum Faradaic efficiency of 69.9 % (10 min)-2 V, BG dye removal 95.1 % (5 min)-2 V in KHCO $_3$  electrolyte solution. The studies will be helpful in water purification with maximum CO $_2$  reduction for our future energy applications.

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