Recent Advancements in Electrochemical Conversion of Carbon Dioxide

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Electrochemical reduction of carbon dioxide into eco-friendly and clean products is a promising approach to eradicate pollution. Although carbon dioxide emission is inhibited by the advent of renewable sources of energy, it is present in the atmosphere and needs to be cleaned. The reduction of carbon dioxide from atmospheric gases can be accomplished by its adsorption and subsequent transportation to electrolytic chambers, where it is reduced to hydrocarbons, organic acids or carbonates. This review focuses on developing a three-compartment electrochemical cell to reduce carbon dioxide used as a catholyte. Various factors affecting the electrochemical reduction of carbon dioxide and recent advancements in this field are deliberated.

**Keywords:** Carbon dioxide, Formic acid, Electrochemical conversion, Organic acids.

1. INTRODUCTION

With the growth of the population, there is a rapid increase in the demand for vehicles, these vehicles mostly run-on fuels and some are powered by electricity. The increase in the use of fuel-based vehicles is the primary reason for the rise in carbon dioxide (CO_2) in the atmosphere. Apart from vehicles, various iron and steel industries which run on coal, their by-product includes gases like carbon dioxide, nitrogen, oxygen and carrier gases, therefore altogether the carbon footprint of the environment increases. There are various methods to reduce the CO_2 percentage in the atmosphere such as (i) capturing and compressing the combustion exhaust (i.e., flue gas), (ii) transporting the captured product through pipelines, and (iii) utilizing it in various industries such as beverage industries, urea production, enhanced oil recovery and dry ice production [1]. Moreover, CO_2 can be used for dehydrogenation reactions like the selective dehydrogenation of ethylbenzene, the product formed is styrene, additionally, it can also dehydrogenize butene, propylene, and ethene [2]. Apart from industrial uses of CO_2 for making beverages, urea, or any bio-degradable product, the electrochemical reduction of CO_2 can also generate electricity. It is possible by electrochemically reducing CO_2 without the reduction of H_2O to H_2 [3].

CO_2 is a thermodynamically stable molecule; therefore, a single electron reduction reaction should take place to convert linear CO_2 molecules to bend CO_2^- molecules. Such conversions require high thermodynamic energy $E_0 = -1.92$ V, which would be provided through an electrochemical cell.

In order to carry out any process over CO_2, CO_2 should be first captured from the atmosphere, therefore various routes have been developed to capture CO_2. By such routes, it can either be first absorbed and then cryogenically distilled, or must be adsorbed, then subjected to chemical loop combustion and finally separated by a membrane or using hydrate-based separation [3].
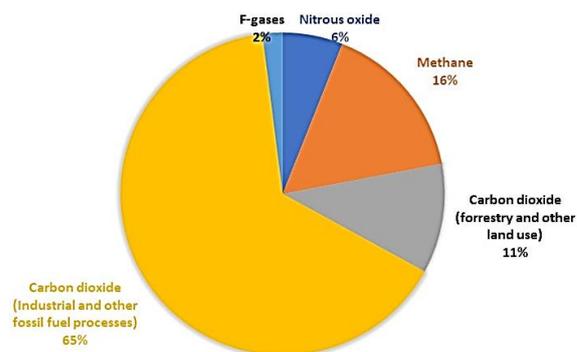
Fig. 1 – Global greenhouse gas emission (reproduced from IPCC 2014)

For the utilization of CO_2 in the above-mentioned industries, CO_2 should be reduced. The electrochemical reduction of CO_2 is carried out by reacting it either with water or any organic solvent to convert it into desirable products like methane, ethane, formic acid, and other carboxylic acids. Numerous catalysts have been developed which are used as a cathode material to increase the efficiency of the electrochemical reduction of CO_2, these catalysts include copper, silver, gold, tin, Fe(0) porphyrin, platinum, palladium and nickel. Catalysts such as Au, Ag, Ni and Fe(0) porphyrin enhance the formation of carbon monoxide while inhibiting the formation of hydrogen. On the other hand, Pt catalysts are used for the conversion of CO_2 into methane and are usually used in the form of Au-Pt bimetallic compounds, where Au is used to speed up the production of carbon monoxide. Moreover, there are catalysts such as Pd, which help in the electrochemical convertion of CO_2 to carbon monoxide and carboxylic radicals. However, their catalyzing

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efficiencies are further improved by using electrodeposition or taking nanoparticles of high activity materials, as these active sites act as a promoter, where CO_2 is adsorbed and its symmetry is changed to convert a neutral molecule to a highly reactive ion [4].

Nowadays, advancements take place wherein the efficiency of the electrochemical reduction of CO_2 can be increased by using ionic liquids as the anolyte material. The interesting fact regarding ionic liquids is that it is a highly reactive polymeric solid material, which reacts with CO_2 to increase faradaic efficiencies of CO and H_2O, wherein an active site as such is not necessarily required. Such ionic liquid materials are 1-allyl 3-methyl imidazolium chloride (AmimCl), poly(2,6-dimethyl-1,4-phenylene oxide) (PPO), poly(arylene ether) ketone, poly[1-(2-methacryloyloxy)ethyl]-3-butyl imidazolium cations etc [5]. The electrochemical reduction of CO_2 can be powered by renewable electricity such as wind, geothermal and solar.

This review article aims to demonstrate various factors affecting the electrochemical conversion reaction and recent advancements in the field of electrochemical CO_2 reduction.

2. CONSTRUCTION OF AN ELECTRO-CHEMICAL CELL

A three-compartment electrochemical cell comprises catholyte and anolyte chambers and is attached with gas diffusion electrodes [6]. CO_2 is taken as a catholyte material because the reduction of CO_2 takes place over the cathode. Therefore, the use of nanoparticle catalysts (such as Sn, Pd) or active materials (Au, Ag) in cathode materials would increase the grain boundary energy of the surface, acting as a promoter to enhance the CO_2 reduction reaction. The resulting product corresponds to the taken anolyte, therefore, when obtaining formic acid, deionized water is taken as the anolyte [6], and while maintaining the same electrolyte and replacing the electrode with a carbon nanospike with electronucleated Cu, the resulting product is methane along with CO, formic acid, and ethylene [8]. Serpentine flow field models are used to circulate the electrolyte to increase the efficiency of the reduction reaction.

2.1 Working Principles

Three compartments of the electrochemical cell consist of a cathodic layer with pH varying from 7 to 11, the central compartment with pH stretching up to 7 and an anionic ionomer. The electrochemical conversion of CO_2 into acid ions begins with the reduction of the chemical present in the anode compartment like the conversion of deionized water into H^+ ions when deionized water is used as the anolyte [9], 1-ethyl 3-methyl imidazolium tetrafluoroborate into 1-ethyl 3-ethyl imidazolium ion [10]. The cathodic compartment is occupied by CO_2 gas, which passes through the gas diffusion electrode layer /membrane interface to interact with the H^+ ion evolved through the protonation of the deionized water molecule (Fig. 2). The movement of CO_2 is enhanced by serpentine flow field attached with cathodic compartment; it is because of the high gas flow rate arising due to the pressure difference created by the serpentine channels. CO_2 reacts with the H^+ ion or 1-ethyl 3-methyl imidazolium

ion [EMIM]$^+$ to form formic acid or [EMIM]$^+$[CO_2]- respectively.
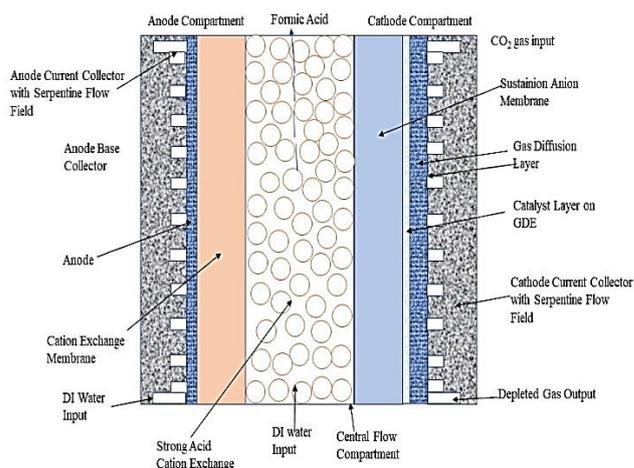

Fig. 2 – Three-compartment cell configuration [6]

Table 1 – Various investigations of the CO_2 reduction reaction on different cathodes

Cathode	Anode	Faradaic efficiency	References
Toray paper with 50 % PTFE proofing	IrO_2 nanoparticles sprayed over PTFE electrode/titanium-based electrode	47 % (at 0.05 mL/min), 88 % (at 0.5 mL/min)	[6]
Copper	Carbon nanospike	71 %	[8]
Copper plates	Fe (II)	66 %	[7]
Nanowires-tin oxide	Carbon black composite	87.40 %	[9]
Tin sheet	Ti/IrO_2-Ta_2O_5 sheet	35 %	[13]
Polydopamine with a catalyst enzyme (E, FDH)	Nanoporous cobalt phosphate/bismuth vanadate	Nearly 100 %	[14]
Silicon	Copper		[15]
Fe^0 porphyins based metal complexes	Cobalt	80 %	[16]
5,10-di(2-napthyl)-5,10-di-hydrophenazine)	Iron(II)	90 %	[17]

The electrolytic reduction reaction generally takes place over the cathode surface and the reaction is enhanced at high-energy surfaces such as grain boundaries or when nanoparticles are used as a catalyst in the cathode or when the cathode is electrodeposited with another material. Upon electrodeposition grain boundaries of the cathode material get activated, hence promoting the reduction reaction.

Table 1 shows various studies which have been carried out on CO_2 reduction reaction over the copper cathode. Moreover, an increase in faradaic efficiency (efficiency associated with the charge transfer in a system causing an electrochemical reaction) has been observed when copper foams are electrodeposited over copper skeleton over the copper-based electrode. There are certain conditions upon which the electrochemical conversion of CO_2 depends. 1. The parameters such as current density, cell voltage, and electrolytic composition control the electrochemical synthesis of the final product. 2. The

cell temperature ranges between 295 to 300 K as well as the standard pressure is maintained during the electrochemical conversion reaction. 3. The catalyst layer acts as a good adhesion to the cathode surface [11, 12].

3. FACTORS AFFECTING THE OPERATION OF AN ELECTROCHEMICAL CELL

In a 3-compartment electrochemical cell, the catalyst is used on the cathodic compartment to increase the efficiency of the CO_2 reduction reaction. These catalysts are used as high-energy sites wherein straight CO_2 molecules are adsorbed and energetically converted into CO_2^- bent molecules. The catalysts can be categorically divided depending on the product formed such as (i) In, Sn, Hg and for the selective production of formate; (ii) Ag, Au and Zn are used for carbon monoxide as evolution; (iii) hydrogen evolution reaction can be catalyzed by Fe, Pt, Ni and Ti; (iv) hydrocarbons and oxygenates production can be catalyzed by Cu or copper-oxide derived Cu [18]. This review work undergoes the advantage of using different catalysts, which have been described below.

Au catalyst: Pure copper used as a cathode shows the lowest electrochemical activity; however, upon addition of Au nanoparticles, $CuAl_3$ is formed showing the highest activity in the series. It has been found that faradaic efficiency for CO increases when the Au content increases and faradaic efficiency for H_2 decreases when the Au content increases. Therefore, where a hydrogen atom binds to carbon during the reaction pathways, faradaic efficiency for their final products decreases, following a similar trend for H_2, while faradaic efficiency of CO shows the opposite trend [19].

Ag catalyst: Ag catalyst can be used by electroplating/electrodepositing it over Cu films, known as Ag-IO (silver inverse opals) electrodes. Depending on roughness, porosity, and thickness of Ag films over the cathode, the CO concentration can be varied. Yoon et al. carried out an experiment showing the effect of faradaic efficiency of CO upon varying the applied potential of Ag film and found that faradaic efficiency for CO formation increases with increasing the thickness of Ag electrodes [20]. Faradaic efficiency for CO formation also depends on displacement time apart from thickness. Lee et al. carried out an experiment demonstrating the CO faradaic efficiency of Ag/Cu foams with respect to the displacement time and found that within the displacement time ranging from 20 to 300 s, the CO faradaic efficiency is ~ 10 % and with increasing the displacement time to 600 s, faradaic efficiency increases to 29.4 % [21].

Sn catalyst: Sn catalysts are plated over the Cu shell in a ratio such that 0.8 nm of Sn is plated over 7 nm of Cu shell, this yields a Cu/Sn core/Sn structure, further getting oxidized to SnO_2. Li et al., in their experiment, found that with increasing the thickness of the SnO_2 shell structure, the major product results to be formate ion [22]. Although the major product is formate ion, its faradaic efficiency decreases with increasing the potential difference of Sn foil. Moreover, faradaic efficiency of the formate ion formation is increased with growing Sn dendrites. Therefore, a significant increase in faradaic efficiency from 49.1 to 56.1 % upon using Sn dendrites over Sn foil is observed at a constant potential difference

of 1.36 V [23].

Fe(0) porphyrin catalyst: Upon using Fe(0) porphyrin catalyst over the cathode, the main reaction product formed is carbon monoxide and the formate ion as the side-product [24]. Dong et al. demonstrated that the faradaic efficiency of carbon monoxide formation increases with increasing cathode potential [25].

Pt catalyst: Pt is used as a catalyst for the gas diffusion layer, and it is used when the desired product of the CO_2 reduction reaction is methane. Therefore, Hara et al. noticed that there is an increase in the faradaic efficiency of the methane formation upon the rise in the current density [26]. The performance of Pt catalyst is enhanced upon using depositing Au-Pt bimetallic films over the gas diffusion layer by the magnetic sputtering co-deposition technique. Au and Pt have separate characteristics i.e., Au increases the faradaic efficiency for carbon monoxide formation, and on the other hand, H_2 gas evolution, as well as the formation of alkanes, is supported by Pt cathode [27].

Pd catalyst: Pd catalyst promotes the formation of formate ion through the reduction of CO_2. This reduction reaction can be ascribed to the generation of tensile strain because of the adsorption of key $COOH^*$ as a result of shifting up of the *d*-band center [28]. As the catalyst acts as a high energy site at the cathode, where the initiation of the reduction takes place, it is observed that during the reduction of CO_2, the Pd catalyst is converted into the PdH layer and the $CO^*$and $COOH^*$ radicals are adsorbed over the PdH thin film layer [29].

Ni catalyst: Ni catalyst alone does not affect the electrochemical reduction reaction, but when carbon and nitrogen are dopped over it, the CO and H_2 evolution is enhanced. In the case of nitrogen and carbon doped nickel, there is double layer capacitance developed with one layer developed against CO gas evolution and the other towards hydrogen evolution reaction [30]. Ma et al. further showed that although the addition of nickel does not affect the CO and H_2 evolution, the nickel addition to the nitrogen-dopped interconnected carbon increases the activity of electrochemical reduction of CO_2 [31]. Moreover, the sites where Ni-N is located show the highest selectivity for the electrochemical reaction of CO_2 reduction reaction compared to the cathode.

4. RECENT ADVANCEMENT

In the previous sections, it has been discussed that the part which is used for increasing the efficiency of the three-compartment electrochemical cell is the active site over the cathode, where the CO_2 molecule gets adsorbed and prepared for the electrochemical reduction. Advancement in the field of electrocatalysts takes place to prepare electrocatalysts of complex compositions, such that the number of active sites is increased. Moreover, various metal oxide frameworks (MOF) of high stability and conductivity inhibit the electrochemical reduction process [32]. Recently, MSCC-ET (molten salt CO_2 capture electrochemical transformation) process has been developed, where CO_2 is captured by the molten salt, this promotes the transfer of ions and reactants throughout the cell, further balancing its temperature [33]. These molten salts are oxides of Li^+/Ba^{2+}/Ca^{2+}. The electrochemical reduction process carried out in the MSCC-

ET process is that the O^{2-} ions obtained from the molten salt used as an electrolyte absorb CO_2 and transform into a CO_3^{2-} ion, further reacting with analytical ions to form the reduction products.

Recent studies have shown that even nanoparticles like carbon nanotubes and carbon nanofibers can be grown over the cathode (made up of Fe) in the presence of a Ni-based anode. The formation of a carbon nanotube over the cathode is carried out by deposition of carbon over the cathode due to electrolytic dissociation of Li_2CO_3. It was found that multi-walled carbon nanotube can be obtained through such techniques, and the internal diameter as well as the number of walls of the nanotube depend on the erosion of the anode, which acts as a catalyst i.e., the smaller the size of eroded anode particles, the larger the diameter and the lower the number of walls of the multi-walled carbon nanotube [34].

Nowadays, cathode materials are no more restricted to Cu or any active metal electrodeposited or used as a nanoparticle catalyst over Cu; however, developments have reached a point wherein a nanoporous PTFE membrane can be used as electrocatalyst support. Clark et al. conducted the reduction of CO_2 experiment using Ag-deposited over nanoporous PTFE as a cathode material. The sites of Ag atoms over the PTFE cathode act as high-energy sites to absorb and electrochemically activate CO_2, so that the electrochemical reduction is carried out with the evolvement of H_2 gas, CO gas and formic acid as the end products [35]. Innovation took height, Huan et al. demonstrated a technique through which they carried out the electrochemical reduction of CO_2 by pyrolyzed Fe-N-C material. Different combinations of Fe-N-C have been derived, out of which FeN_4 moieties are seen to be as the active sites for carrying the electrochemical reduction [36].

Although Cu is a prominent material for catalyzing the electrochemical reduction of CO_2, there remains the problem of high overpotential (the potential difference between the electrode and electrolyte surfaces at their interface) developed, while carrying out the reaction. It has been noticed that > 500 mV of overpotential is required to generate the current of a current density of 1 mA/cm^2, therefore new alloys such as Au-Pd, Pd-Pt, Ni-Ga have been developed as materials for the cathode in place of Cu. As a result, Torelli et al. came up with a method through which products such as CH_4, C_2H_4 and C_2H_6 can be produced by electrochemical reduction of CO_2 using Ni-Ga alloy as a cathode material [37]. It has been shown in Fig. 1 that a GDE (gas diffusion electrode) layer lies on both sides of the electrochemical cell i.e., on the cathodic part and the anodic part. The GDE layer consists of a cathode fiber substrate (CFS) and a catalyst layer (CL). This experiment introduces the use of a microporous layer (MPL) to act as an interface between CFS and CL. It has been seen that the electrochemical cell lacking MPL shows drastically low current density, even lesser than 30 mA/cm^2. This could be because MPL helps in distributing irregularly disturbed cathode particles in between carbon fibers of CFS [38]. Subsequent research was carried out and boron phosphide nanoparticles were found to replace Cu as it increases the current density for electrochemical reduction of CO_2 [39]. All such improvements have been shown in the field of electrochemistry of CO_2 reduction.

5. CONCLUSIONS

This review describes several cathodic materials which could be used for the electrochemical reduction of CO_2. With Cu as the primarily used cathode material, this study goes on to explain how the nanoparticle deposition over the Cu cathode could accelerate the process of CO_2 reduction. Apart from Cu-based cathode, this article demonstrates the advancement in the field of electrochemical reduction reaction of CO_2 with the use of ionic liquids or $Na^+/Li^+/Ba^{2+}$ oxides as the cathodic material. The reduction of CO_2 is carried out with the formation of formic acid, methane, ethane, and carboxylic acids as the end product. Moreover, advancements have led to the development of nanostructures of carbon such as carbon nanotubes and carbon nanofibers after the electrochemical reduction of CO_2. Additionally, issues with Cu as a cathode such as an overpotential have been discussed.

The electrochemical conversion of CO_2 is not only limited to a small scale, but the establishment of the solid oxide electrolyzer cell (SOEC) has scaled up its electrochemical conversion.

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Останні досягнення в електрохімічному перетворенні вуглекислого газу

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Електрохімічне відновлення вуглекислого газу в екологічно чисті продукти є перспективним підходом до викорінення забруднення. Хоча викиди вуглекислого газу гальмуються появою відновлюваних джерел енергії, він присутній в атмосфері та потребує очищення. Відновлення вуглекислого газу з атмосферних газів може бути здійснено шляхом його адсорбції та подальшого транспортування в електролітичні камери, де він відновлюється до вуглеводнів, органічних кислот або карбонатів. Стаття зосереджена на розробці трикамерної електрохімічної комірки для зменшення вуглекислого газу, який використовується як католіт. Розглядаються різні фактори, що впливають на електрохімічне відновлення вуглекислого газу, і останні досягнення в цій галузі.

Ключові слова: Вуглекислий газ, Мурашина кислота, Електрохімічне перетворення, Органічні кислоти.