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Magnesium Oxide NPs Carbon Paste Ion-Selective Electrode Industrialization and Potentiostat Cyclic Voltammetry Detections for Determination of Mg+Ion in Various Solutions

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ABSTRACT

A modified ion-selective electrode with graphene powder and Magnesium Oxide Nanoparticles, a newform highly sensitive paste and selective to electron motion in redox process by cyclic voltammetry Potentiostat technical applied. A sol-gel bottom-up nanoparticle synthase method used to prepare MgO-NPs, SEM and XRD appear 18 nm average particles size, carbon powder with paraffine oil drops and MgO-NPs mixed to form homogenous black paste. Full one end glass cylinder with 0.4 r2 dimeter with copper wire to form a working electrode, plamSens 4 Potentiostat cyclic voltammetry used to motion redox processing in cell reaction by link calomel reference electrode and platinum wire axillary electrode and working electrode. 0.1 v/s, (± 2E) V parameters select, (10-100 ppm) standard and working Magnesium solutions a trace concentration used in this study. Epa/Epc > 200 V with hydrogen over potential refer to a reversable CV mechanism, kinetic and thermodynamic were study with (293-333) K the negative signal shifting in cyclic voltammograms, Randles-Sevcik equation used to found Diffusion coefficient equal 3.6 × 10-5, and rate constant, calibration curve range 1.2 × 10^{-3} and 10 ~ 100 ppm respectively.

1. INTRODUCTION

Magnesium oxide nanoparticles are considered one of the most important molecules in modern research, as they have recently attracted the attention of scientists and researchers due to their many uses in various fields, including medical, diagnostic, therapeutic and conductive, due to the distinctive chemical properties of these molecules [1]. As is known in our current world, the uses of magnesium oxide nanoparticles are in antioxidants, antibiotics, cancer treatments and antibacterial. Modern science focuses on nanoparticles until they have become a main focus in most areas of modern research due to their unique properties and wide applications [2]. The large surface area of nanoparticles is one of the most important physical properties of nanoparticles, which makes them possess unique properties, including magnetic, optical, electronic, mechanical and electrical conductivity properties [3-6]. The methods used in the synthesis of nanoparticles, including physical, chemical and green methods, are considered among the most important factors in determining the size and properties of the particles [7, 8]. Metal oxide nanoparticles are of great interest to researchers due to their wide range of uses due to their unique physical and chemical properties and their various applications in various fields such as sensor technology and sensors, especially in high-precision medical devices, as well as in science, environment and pharmaceutical compounds [9-13]. Nano magnesium oxide is distinguished from other oxides of different elements and has received great attention from the scientific and research community due to its non-toxic nature, biocompatibility, high chemical stability and resistance to various conditions. The US Food and Drug Administration has considered it a safe substance for human consumption [14, 15]. The ionic nature of MgO-NPs, large specific surface area and distinctive crystalline structure allow easy interactions and simple interaction mechanisms with various systems, including biological, chemical and electrical, which made it have various uses, for example, catalysts, disinfectants, sensors, sensors, super-sensitive conductors and thermal resistance. Based on all of the above, focusing on MgO-nanoparticles is of utmost importance [16]. From the superconductivity of nano magnesium oxide, we start to create and manufacture highly sensitive ion selective electrodes. By mixing it with graphite powder to form a homogeneous paste that conducts and selects magnesium ions in different solutions, including pharmaceutical, aqueous, blood, etc. [17]. In other hand, graphite powder wide used in electrochemistry for several reasons such as high electrical conductivity, thermal conductivity, chemical stability, Lubricity, Porosity and Surface Area, Cost-Effectiveness, Ease of Fabrication and Corrosion Resistance [18, 19]. To monitor the work of the ion selective electrode, the use of Potentiostat is very easy to follow the movement of electrons resulting from the oxidation and reduction process of ions in their solutions and monitor the

different mechanics of electron flow in the electrical circuit of the galvanic cell [20]. Cyclic voltammetry is one of the applications of Potentiostat in electrochemistry. It is the ideal choice for enjoying the oxidation and reduction process through cyclic voltammograms data, through which all variables, physical and chemical properties, as well as concentrations in solutions are calculated [21].

2. EXPERIMENTS

2.1 Synthesis of MgO NPs

Sol-gel methods used for synthases MgO-NPs by dissolving 0.2 mol Mg $(NO_3)_2.6(H_2O)$ in 500 mL D.W with pH 9.45 by adding base solution (NH_3) 20%. In other hand, salt hydrolysis causes the pH of the solutions decrease. Base solutions adding by titration system with controlled drop-wise 1drop/2min. The gel material (Mg $(OH)_2$) was generated when the solution was placed on a Hotplate Stirrer and stirred for an hour at 80℃ at a moderate pace. After cooling to room temperature, the resulting material was filtered and given more than five washes with deionized water. The final product was obtained by calcining the resultant solid for 2.2 hours at 500℃ after it had been oven-dried for a whole night at 100℃ [22].

2.2 Electrochemical application

Potentiostat (CV) application, (0.05, 0.1, 0.2, 0.3, 0.4 and 0.5) V initial and final linear voltage applied on the working electrode, cyclic voltammograms detected and mutation a response current that happened with redox process. The oxidation and reduction process produces a flow of electrons as a result of applying different voltage values, which causes an electrical potential difference to occur on the surface of the working electrode, which in turn causes the resulting current to be recorded. The changes in the currents are monitored as a result of the different conditions applied to the electrical cell through the oxidation and reduction peak signals that appear in the cyclic voltammogram. We can also detect the reaction mechanism resulting from the movement of electrons from the solution to the electrode. KCl the supporting electrolyte used in all experimental study. The range of parameters that used to selected the best condition of working electrode detection. The potential Ea, Eb and Ec (start, end and binging) (-1.0, 1.0 and -0.1) respectively. And scan rate (0.05, 0.1, 0.2, 0.3, 0.4 and 0.5) $v.s^{-1}$, 0.5M KCl supporting electrolyte with pure magnesium chloride solution 0.1 M. also the time that applied for equilibrium state equal to 30 second. The working electrodes' surface was polished with fine paper and then cleaned with double-distilled water to create an inert atmosphere for all of the experiments. This was accomplished by purging the cell solution with nitrogen gas for approximately fifteen minutes and keeping it over the cell solutions while the voltammograms were being recorded. An Auto-Lab Potentiostat (palmSens4, Netherlands) and PSTrace Software were used to conduct the electrochemical measurements. A MgO-NPsCP working electrode (Figure 1). Ag/AgCl and Pt-wire were reference and axillary electrodes, respectively, the pH/ion meter used for medium controlled. DW using to create all solutions. Merck provided all of the analytical grade reagents, as well as the graphite powder and paraffin oil. In the pH range of 2.0-8.0, acids and bases were diluted to high concentrations. All solutions were deoxygenated for about 20 minutes with pure nitrogen gas before each electrochemical experiment.

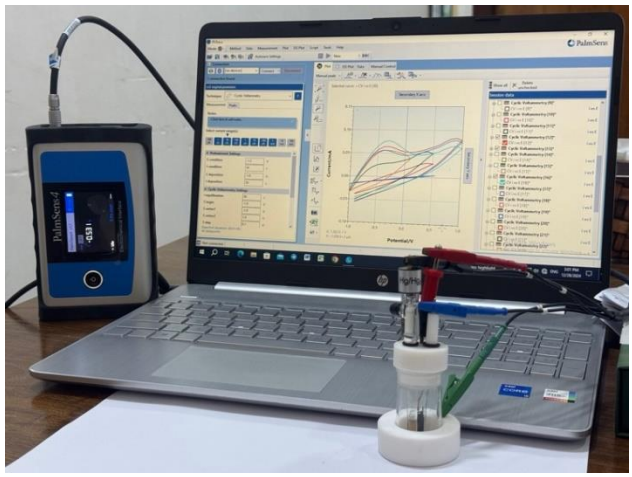

Figure 1. Cyclic voltammetry cell

2.3 MgO-NPsCPE synthases

The MgO-NPs modified graphite paste sensor was form 0.7 g of MgO nanoparticle, 0.3 g of graphite powder and 0.3 ml of paraffin oil until a dark-Gray uniform paste was obtained, the homogenise paste was distributed in the form of discs 3 mm thick and 0.3 in diameter, a soft paper used for surface polished Figure 2 refer to synthetic working electrode.

In this study was used different family of drugs, also used various electrolytes, salt, and stock solution. The stock solutions 0.1M from Mg-ion solution, with MgO nanoparticles graphite paste sensor, calomel and platinum wire was (working electrode), (reference electrode) and (axillary electrode) respectively and contacted with Potentiostat cyclic voltammetry.

Figure 2. MgO-NPs carbon paste working electrode

3. RESULT AND DISCUSSION

3.1 Structure of MgO-NPS

Figure 3 and Table 1 refer to X-RD detection for the commercial and synthases MgO nanostructure. Decrease in

diffraction peaks when temperature increase, the date refers to nanoflakes MgO for both synthases and commercial, in anther hand FCC-structure (1 1 1), (2 0 0), (2 2 0), (3 1 1), and (2 2 2).

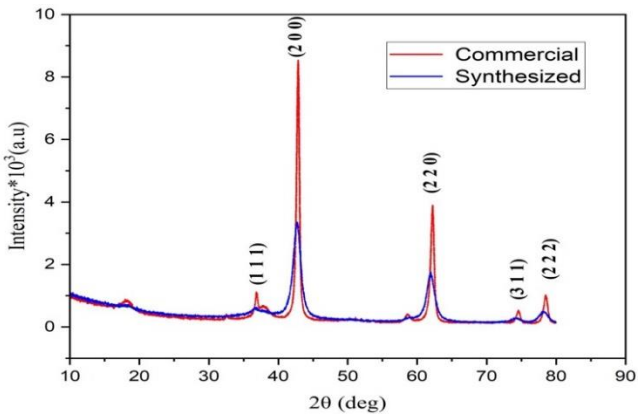

Figure 3. XRD detection magnesium oxide crystal size

Table 1. Debye- scherrer parameters magnesium oxide crystal size

2θ (deg)	Reflection Planes	WHM	Crystallite Size (nm)
35.8	(1 1 1)	0.38	21.0
41.4	(2 0 0)	0.40	18.8
61.8	(2 2 0)	0.53	18.3
73.7	(3 1 1)	0.52	18.3
77.3	(2 2 2)	0.52	17.8
The average crystallite size			18.8

3.2 Metal Oxide Nanoparticles

3.2.1 Magnesium oxide nanoparticles

Figure 4 shows a SEM of agglomerated MgO-NPs with an average size of 18.8 nm.

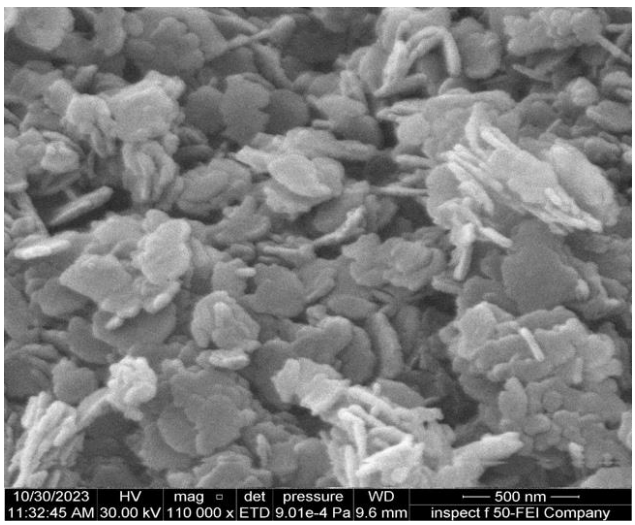

Figure 4. SEM image of MgO nanoparticles

A number of factors contribute to the formation of nanoflakes, nanoflowers, and nanoparticles, including the employment of controlled synthesis processes, response parameter adjustment, post-synthesis treatments, and the careful selection of precursors [23]. With these characteristics, researchers may create unique nanoflakes, nanoflowers, and nanoparticles that can be used in a variety of fields, including electronics, catalysis, sensing, and medical nanostructures. Response factors that affect the reaction rate throughout the nanostructure-making process greatly influence their size and form [24]. Precursor concentrations, temperature, pressure, pH, and reaction time are all included. One strategy to raise the possibility of attaining superior nanostructure development is to improve the reaction time [25]. Scanning electron microscopy (SEM) images of commercial and synthetic magnesium oxide (MgO) nanoparticles show how these parameters directly affect the pace at which nanostructures develop, which in turn affects the size and shape of the structures. According to the data, the particles seemed to have smooth surfaces and a nanoflakes shape. While the majority of the particles were grouped together, others were widely scattered. The average size of the artificial MgO nanoflakes was 22.63 [26]. Figure 4 the size ranges from 18.73 to 29.97 nm, and the shape and dimensions of nm. The commercial MgO nanoflakes, on the other hand, ranged in size from 26.23 to 52.05 nm, with an average of 44.3 nm. Rapid reduction, assembly, and sintering of room-temperature nanoflakes produces the observed nanostructures [27].

3.3 Electrochemical study of MgO-NPs-cp electrode

3.3.1 Conductivity

Figure 5 refer to conductivity experimental by used the conductivity devices (University of Hilla, medical physics laboratory), the result shows a highest working electrode conductor when applied deferent potentials with average (0.1 – 10) V. all result refers to 10% The rate of change in the voltage reading applied and recorded by the detector after passing through the surface of the electrode. This is a sufficient indication of the amount of homogeneity in the dough used and the absence of any amount of voltage being lost, which ensures accuracy in the reading during the various experiments of this study.

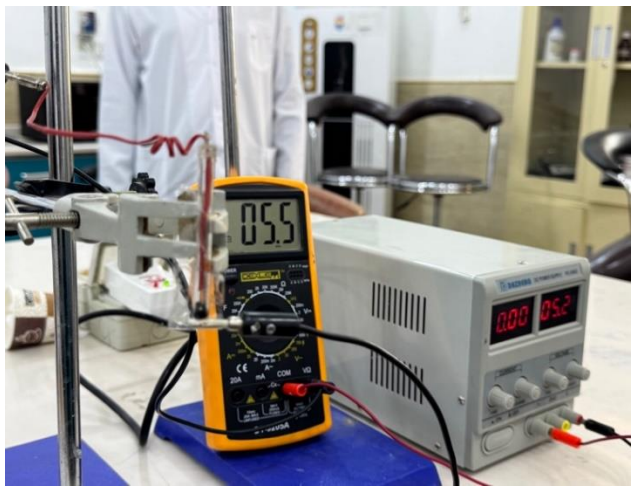

Figure 5. The conductivity experimental

3.3.2 Selectivity of electrode

The signals of redox peaks refer to the electron transport from solution to detector by surface of working electrode. To determine the type of working electrode, the triangular current cell was connected to the Potentiostat and the cyclic voltammetry method was determined as a specific mechanism to monitor the movement of electrons resulting from the chemical oxidation and reduction process during the applied

of voltage to the standard solution. On the other hand, a 0.1 molar concentration of salts of different elements was prepared, including zinc chloride, magnesium chloride, copper chloride and zinc chloride, and standard conditions were used to conduct the selectivity experiment. 0.1 v. s^{-1} was determined for all experiments. The result showed through the cyclic voltammograms the absence of any activity response to the oxidation and reduction process when using salt solutions that differ from the type of electrode paste surface. The detector recorded an oxidation signal resulting from an electron transfer movement when using a magnesium chloride solution. This is considered conclusive evidence of the selectivity of the manufactured electrode, which results from the electronic affinity between the electrode surface and magnesium ions in the transformer with the help of the supporting electrode in carrying weak currents Figure 6 refer to response signals.

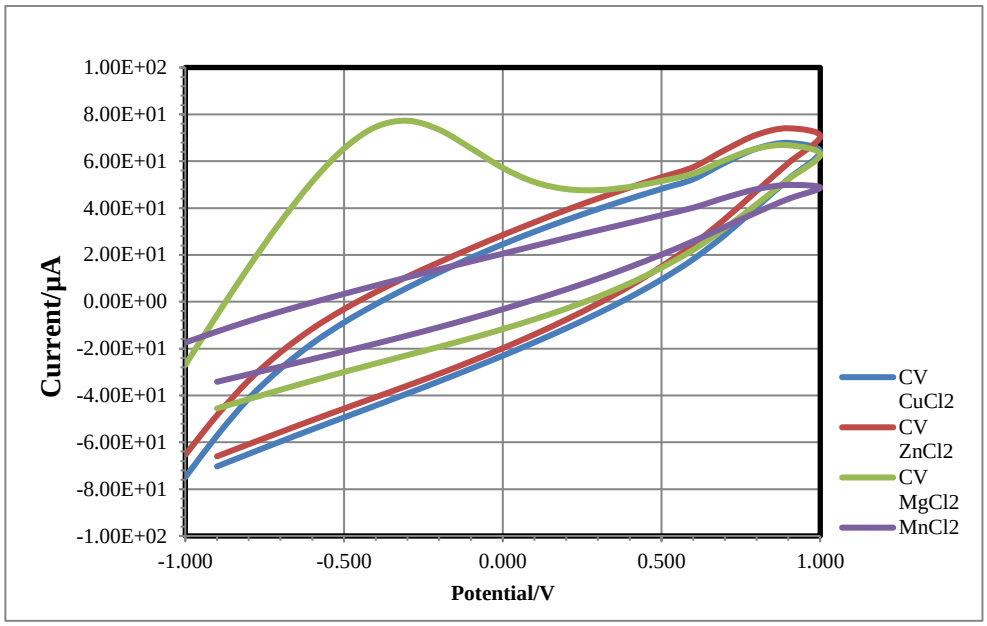

Figure 6. Cyclic voltammograms of MgO nanoparticles electrode in the presence of 0.1M of $MgCl_2$, $ZnCl_2$, $CuCl_2$ and $MnCl_2$ solutions

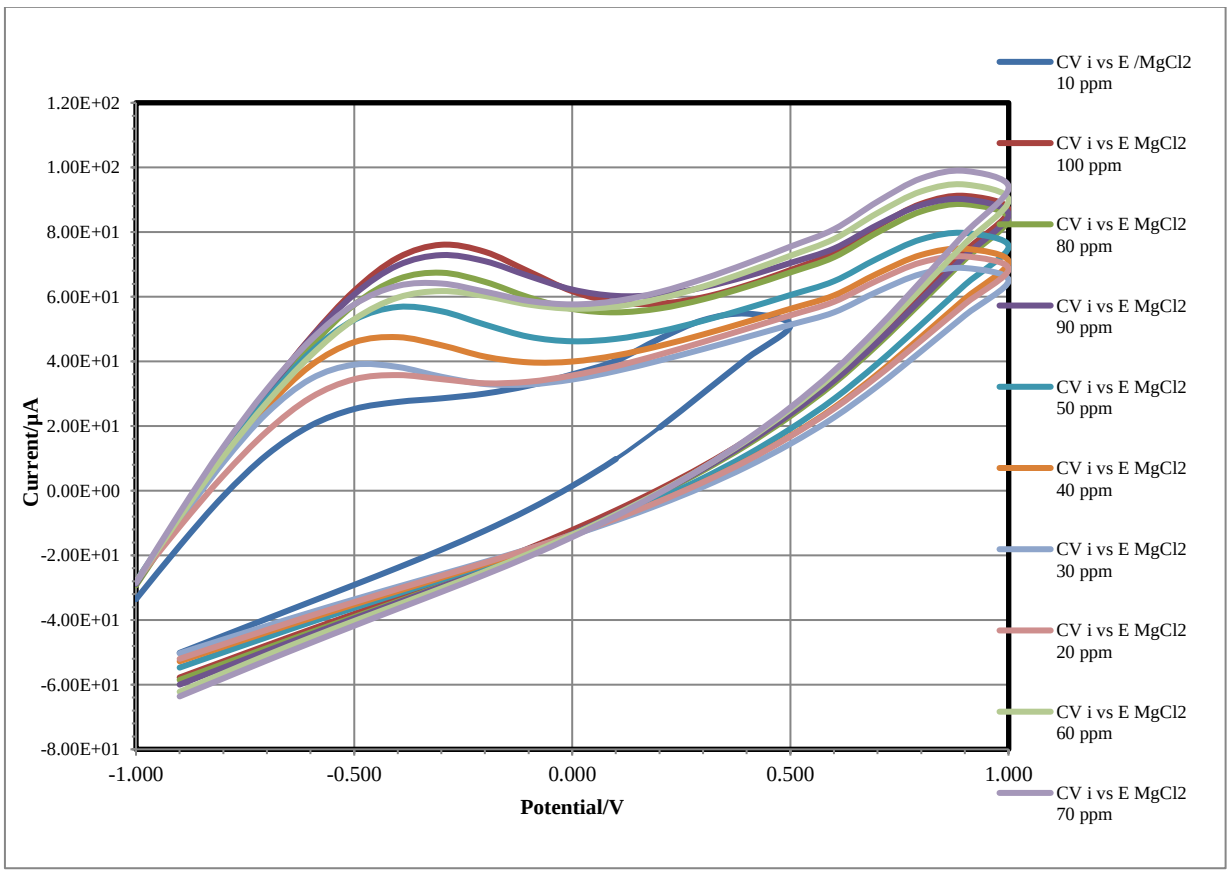

Figure 7. Calibration Curve a cyclic voltammograms of MgO nanoparticles electrode in the presence of (10 – 100) ppm of $MgCl_2$ solutions

3.3.3 Temperature study

Calomel electrode was conducted using by cyclic voltammetry within the temperature range of (20–50) ℃; The fluctuation of the CV-grams curves detect to calculate activation energy for a broad range P-(V) where typical redox peaks occurred. Measurements of stationary current intensity in pure kinetic areas made it possible to calculate the activation energy. The temperature dependence of the charge-transfer resistance and rate during the electro-oxidation process was continuously linked to the surface coverage by a diffusion intermediate. This relationship was evaluated and discussed, demonstrating that electro-oxidation occurred through a complex process lacking significant contacts with the MgO nanoparticle electrode surface [28]. Eq. (1) used to determine Ea.

$$\ln D = \ln Do - (Ea/RT) \quad (1)$$

The free energy was estimated using the slop ln D & 1/Tk

$$\Delta G = - nFR(\Delta Ep) \quad (2)$$

The free Gibbs energy, which related to the spontaneous reaction, Kp by Nicholson equation (Eq. (3)) [29] based on the previous result:

$$K = Ko \exp(-\alpha F/RT) (E - Eo) \quad (3)$$

The first order reaction of cyclic voltammetry is 1.43×10^{-5} S^{-1} for the quasi-reversible process. The physco-parameters for the MgO-NPs CPE are shown in Table 2.

Table 2. Physco-parameteters of electrode

Free Energy	Free Gibbs Energy	Order Reaction	Rate of Reaction
51.22 KJ/mol	47.1 KJ/mol	First	1.12×10^{-3} S^{-1}

3.4 Magnesium calibration plots and determination by used MgO-NPs-CPE

Figure 7 refers to calibration curve CC to the MgO-NPs-CPE, the linearity of the Calibration Curve used to detection of Mg^{+2} limits, were obtained using cyclic voltammetry. A MgO nanoparticle modified electrode was tested for its capacity to differentiate the electrochemical response of Mg^{+2} concentrations. As a result of its improved removal of capacitive background current, CV was utilized for simultaneous species determination. Using a MgO nanoparticle electrode, analytical tests were carried out on various quantities of 0.1 M from $MgCl_2$ pure stock solution. The CC produced by CV at 10-100 ppm by used 10 cyclic voltammetry cells with 25℃ for 10 min for equilibrium after that starting to experimental applied with same conditions for all determination, a clearly reveals the MgO-NPs sensitivity to Mg [30].

4. CONCLUSIONS

When nano metals oxide forms an electrode, it becomes a highly sensitive and selective electrode that detects a trace change in the current running through its solution. As a result, it was employed in the preparation of the electrode as well as the investigation of its physical and chemical characteristics. It was used to measure the quantities of some medicines containing metals. The investigation revealed that shielding cannot be regulated and that an ion selective electrode for one ion alone must be used to prevent redox interactions when a potential is supplied to the reaction cell. The Ipa/Ipc relationship is nonlinear, referring to more than one electron transported at the same time.

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