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The effect of electrolyte concentration on the energy storage using MnO_2 Supercapacitor electrode

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Abstract: Anodic potentiostatic electrochemical deposition was used for constructing MnO_2 amorphous films onto stainless-steel substrates. Effects of concentration of Na_2SO_4 electrolyte solution in the range, from 0.1 to 1 M, on the capacitive behavior, charging/discharging characteristics and electrochemical impedance spectroscopy were studied. The obtained I-V or cyclic voltammogram curves indicate the existence of high capacitive behavior as a good response to the charge carrier's polarizability. The specific capacitance decreases, from 308.52 to 183.9 F/g as the voltage scan rate increases from 10 to 100 mV/s, and the highest maximum and average power obtained are 15 and 4.25 watt which corresponds to 0.5 M electrolyte concentration. Also, the max energy density =50.045 Wh/kg and corresponds to concentration of 0.5 M Na_2SO_4 electrolyte.
With respect to the times of charging/discharging processes, their time constants and specific capacitance are also calculated and their results revealed that the best concentration of Na_2SO_4 among all tested electrolyte solutions to construct MnO_2 based supercapacitor of maximum high capacity (341.99 F/g) is 0.5 Molar. This was interpreted due to the different effect of Na_2SO_4 concentration below and beyond the 0.5 M of electrolyte concentration.
Electrochemical impedance spectra of tested films in the range of 10 mHz to 100 kHz showed both the two resistive and capacitive behaviors which are dependent on the electrolyte concentration noting that lower values of both electrical series and charge transfer resistances are found as 4.9 Ω and 21.4 Ω, associated to the Na_2SO_4 of electrolyte solution of 0.5 M concentration.

Keywords: Anodic deposition; MnO_2 Supercapacitors; electrochemical impedance spectroscopy (EIS)

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I. INTRODUCTION

In the recent past decades, MnO_2 has attracted great attention with respect to the technology of super-capacitor and energy storage. This is due to its linear I-V dependence capacitive electrode, energy storage attributed to ion insertion/desertion within MnO_2surface depending on particle size, surface area and porosity in addition to the abundance and un toxicity of manganese rather than the ruthenium oxide which is toxic and expensive [1-3].For achievement optimized conditions for obtaining high specific capacitance and energy, different morphologies of manganese dioxide have been developed, e.g., nanorods[4], nanosheets[5] and nanowires [6] etc., where, the synthesis route has found to play an important and vital role in determining the required morphology.

MnO_2 has been prepared by several different methods like, chemical precipitate [7], sol-gel [8], pyrogenation[9], mechanical grinding [10], electrochemical deposition [11] and hydrothermal synthesis [10, 12]. It has been reported [4, 11, 14, and 15] that the electrochemical deposition method was proven to be more effective for preparing MnO_2 nanostructures.

Also, electrochemical deposition of MnO_2 could be prepared via two approaches; anodic oxidation and cathodic reduction. In the anodic oxidation, cationic Mn^{2+} precursor is commonly used, but anionic MnO_4^- (Mn^{7+}) is used in cathodic reduction. Both of anodic oxidation and cathodic reduction can be manipulated by potentiostatic (PS) at constant potential as well as galvanostatic (GS) at constant current density processes [6, 16-20].

It is worth mentioning that both (PS) and (GS) techniques could be electrochemically employed to produce nanostructures as well as amorphous films of MnO_2 films. Anodic [21] and cathodic [1] investigations demonstrated that the galvanostatic tested nanostructured MnO_2 films are associated with the higher values of electrical specific capacitance than those ones corresponding to potentiostatic films. On the other hand, the results reported [22] on amorphous MnO_2films, electrochemically and anodically deposited on Stainless-Steel revealed that the (PS) films are accompanied with the higher values of specific capacitance and energy than those corresponding values from the (GS) films.

Anodic deposition of MnO_2 on a carbon substrate in manganese acetate solution was investigated [23], where, the effects of electrolyte concentration, in the range, 0.01 to 1 M and different temperatures from 0 to 50 ^oC, on the characteristics of prepared manganese oxide were explored. The reported results [23] revealed that the concentration of manganese acetate solution could affect the oxidation state of manganese ions as well as the morphology of the deposited oxide, i.e., the specific capacitance of the MnO_2 increased with decreasing deposition temperature as well as the capacitance has its maximum value for the oxide deposited in 0.5 M manganese acetate solution.

The present work is aimed: i) to prepare, anodically and PS electrodeposited MnO_2thin films from 0.25 M $(CH_3COO)_2Mn.4H_2O$ solution at 1 Volt on Stainless-Steele substrate and ii)the effect of molar concentration of the Na_2SO_4 electrolyte on their capacitive behavior for supercapacitor applications will be studied.

II. EXPERIMENTAL

2.1 Electrochemical deposition of MnO_2 films

Stainless-Steel (SS) foils commercially available (type 304) of thickness 0.175 mm were cut as samples of 1× 2 cm^2 each to be used as working electrodes for the electrochemical deposition. The samples were first etched in Hydrochloricacid, 38% concentration for 10 minutes, and then washed with distilled water and air dried. MnO_2 thin films were anodically electrodeposited from 0.25 M $(CH_3COO)_2Mn.4H_2O$ solution by potentiostatic (PS) conditions at 1 Volt. The estimated mass loading of the deposited MnO_2 film was 300 $\mu g/cm^2$, which controlled by adjusting the total charge passed through the electrode during deposition time.

2.2 Electrochemical characterization of MnO_2/SS electrode

All electrochemical deposition and measurements were performed by conventional three electrode system using SP-150 potentiostat/galvanostat device in an electrochemical cell with stainless steel substrate as a working electrode, Ag/AgCl (NaCl saturated) as a reference electrode, Pt wire as a counter electrode, and 0.5 M Na_2SO_4 solution as the characterization electrolyte. The deposited MnO_2 thin films were tested for supercapacitor application by studying the effect of Na_2SO_4 electrolyte concentration in the range, from 0.1 to 1 M on cyclic voltammetry, charge-discharge, and electrochemical impedance spectroscopy (EIS) measurements. Cyclic voltammetry (CV) tests were conducted in a potential range of 0-1 V at scan rates of 10-100 mV/s. Galvanostatic charge/ discharge cycling was conducted at current density of 1-10 mA/cm^2 between 0 and 1 volt. EIS data were collected at alternating current root mean square voltage amplitude 10 mV in a frequency range of 100 kHz-10 mHz.

III. RESULTS AND DISCUSSION

3.1 The cyclic voltammetry curves

The structure of the etched bare stainless-steel (SS) as well as the electrodeposited MnO_2 on it was previously studied using X-ray and SEM imaging [22].

The cyclic voltammetry curves of potentiostatic deposition of MnO_2 films on etched SS electrodes were recorded in an electrolyte solution of 0.5 M of Na_2SO_4 concentration are recorded at different scan rates, ranging from10 mV/s to 100 mV/s in the potential range, from 0 to1 volt are shown in Fig.1. These curves show that, as the electric potential (V) reaches one volt (maximum value) the current (I) start to decay to reach minimum value as the electric potential (V) reaches zero. The growth and decay I-V curves, are forming a nearly rectangular loop of area representing, the stored electric energy as a result of MnO_2 polarization. The obtained curves in this figure 1 indicate the existence of high capacitive behavior as a good response to the charge carrier's Polarizability.

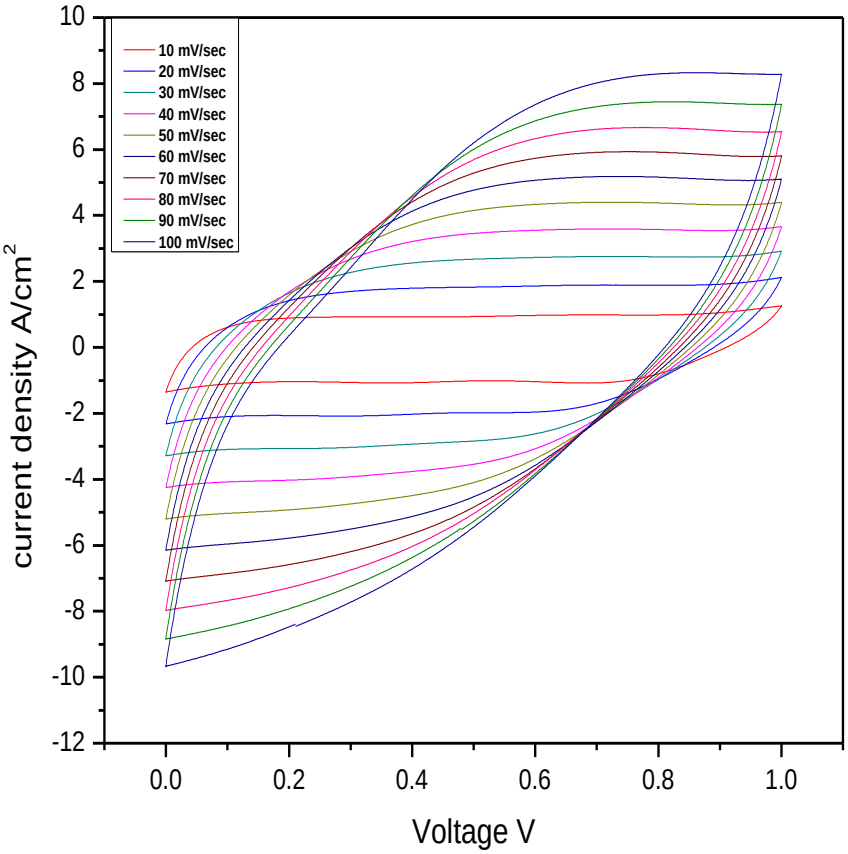

Fig. 1: I-V curves of (PS) deposited MnO_2 films at different scan rates in a 0.5 M electrolyte of Na_2SO_4.

It is well known that, as the integrated area of the I-V curves increases, the higher specific capacitance (SC) of the deposited film can be calculated using the relation S.C = $\frac{Q}{m\Delta V}$,where, (Q) is the capacitive charge, (m) the mass of the deposited film, (ΔV) is the width of the potential window. The values of SC were tabulated in Table (1).

Table (1): Specific capacitance values for tested PS deposited MnO_2 films as calculated at different indicated voltage scan rates.

Voltage Scan rate(mV/S)	10	20	30	40	50	60	70	80	90
Specific Capacitance (F/g) for (PS) method	308.52	278.53	258.77	243.2	230.03	218.68	208.38	198.52	190.67

Refereeing to Table (1), it can be generally observed that, the specific capacitance value decreases as the voltage scan rate increases. This decrease in the SC values with increasing voltage scan rate has been commonly observed. This has been attributed to an increase of the resultant electric field as a result of the polarization of deposited MnO_2.

3.2 Effect of electrolyte concentration on CV curves.

Fig. 2 shows all the cyclic voltammetry (CV) comparison curves of PS deposited MnO_2 films on etched SS electrodes recorded for different molar concentrations, (0.1, 0.3, 0.5, 0.7, 1) in Na_2SO_4 electrolytes, respectively, over the potential range from 0 to 1 V and at scan rate of 90 (mV/S).

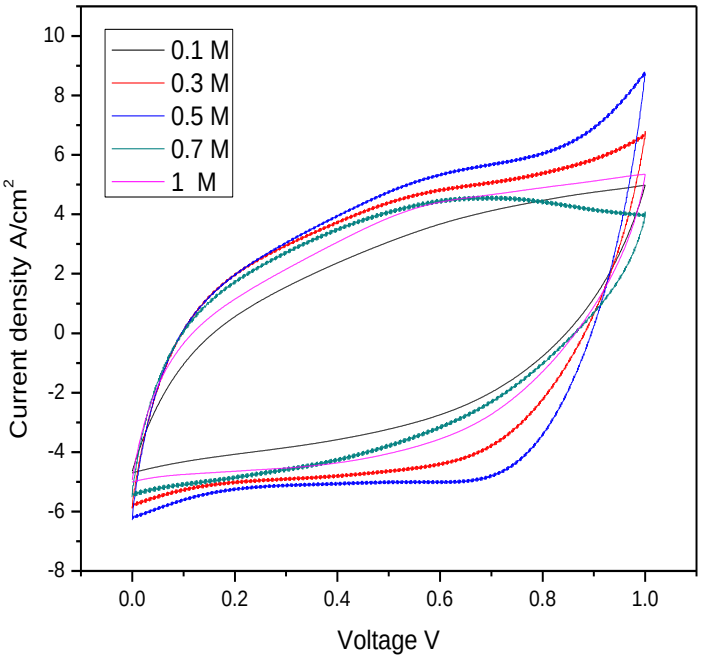

Fig. 2: I-V curves of PS deposited MnO_2 films measured in different concentrations of Na_2SO_4 electrolytes at same scan rate, 90 (mV/s).

From previous I-V curves in Fig.2, the values of the maximum and the average stored electric power was calculated for all tested concentrations of the Na_2SO_4electrolyte and are given in Table (2). However, the variation of the maximum power with the electrolyte concentration is illustrated in Fig. 3.

Table (2): Maximum and average power for tested PS deposited MnO_2 films for different electrolyte concentrations.

Concentration (M)	P_{max} (Watt)	P_{av} (Watt)
0.1	10	3
0.3	13	4
0.5	15	4.25
0.7	9	3.25
1	10	3

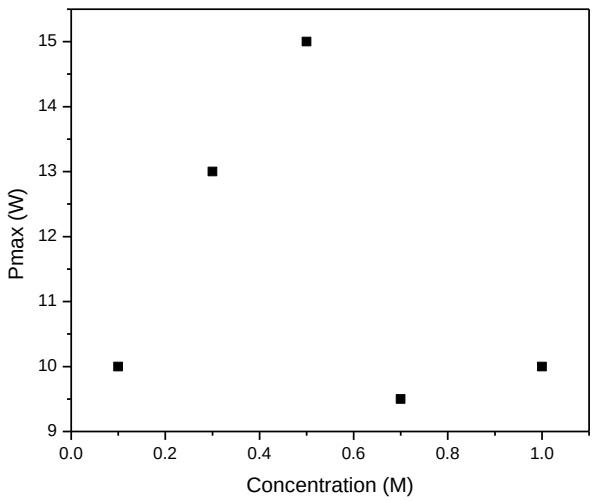

Fig.3: Variation of stored electric power with the concentration of Na_2SO_4 electrolyte.

It is clear from Fig.3 that the highest maximum power obtained is 15 watt which is corresponding to 0.5 M electrolyte concentration. Also, the specific capacitance for each electrolyte concentration was calculated as a function of the voltage scan rate and given in Table (3).

Table (3): Specific Capacitance for tested PS deposited MnO_2 films with scan rate in different electrolyte concentrations as indicated.

Voltage Scan rate (mV/S)	SC (F/g) For (0.1M)	SC (F/g) For (0.3M)	SC (F/g) For (0.5M)	SC (F/g) For (0.7M)	SC (F/g) For (1M)
10	282.225	307.975	360.35	308.325	279.725
20	254.45	288.45	342.6	284.65	270.65
30	236.675	275.125	333.3	268.6	253.25
40	223.25	265.175	317.15	256.425	240.75
50	211.3	256.775	298.3	246.025	230.5
60	200.825	244.725	288.775	236.925	221.775
70	191.75	242.2	280.3	229.15	214.1
80	181.9	235.75	273.275	210.825	207.225
90	173.4	226.75	262.05	207.725	201.2
100	166.75	224.9	249.675	194.6	196.375

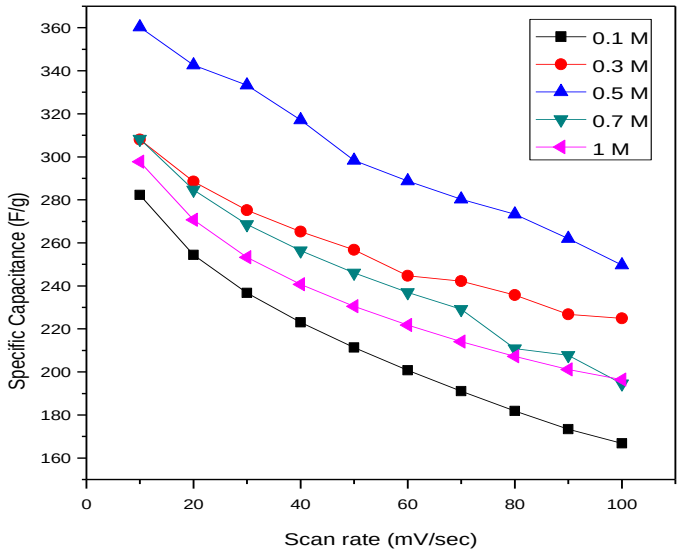

Fig.4: Variations of Specific Capacitance with the voltage scan rate for different tested concentrations, 0.1, 0.3, 0.5, 0.7 and 1 Molar of Na_2SO_4 electrolytes.

It is clear from Fig. 4, for each electrolyte concentration that the specific capacitance values decrease as the voltage scan rate increases. This common behavior may be attributed to the increase of the actual area of the studied amorphous MnO_2 film as has been deposited in a form of hills and valleys [22] with surface area wider than the original electrode area (1 cm^2).

On the other hand, the specific capacitance for each electrolyte concentration as a function of the voltage scan rate (see Table (3)). The recorded maximum specific capacity of the constructed super capacitor was corresponding to 0.5 M electrolyte concentration. This may be due to the increase of the electrolyte molecular polarization till it reaches 0.5 M. After this 0.5 molar concentration, the value of the molecular polarization decreases due to the increase of the electrolyte viscosity, which decreases the molecular orientation, i.e., decrease the molecular polarization and as a result does the specific capacitance.

The values of energy density were calculated for the cases of tested concentrations (0.1, 0.3, 0.5, 0.7, 1) molar of Na_2SO_4 electrolytes by using the equation: $E = (½)\ CV^2$, where C is the specific capacitance and V is the voltage window and listed in Table (4).However, the concentration dependence of that energy density is depicted in Fig. 5.

Table 4: Energy density for tested PS deposited MnO_2 films for different electrolyte concentrations.

Concentration(molar)	Energy density(Wh/kg)
0.1	39.19
0.3	42.77
0.5	50.045
0.7	42.82
1	41.35

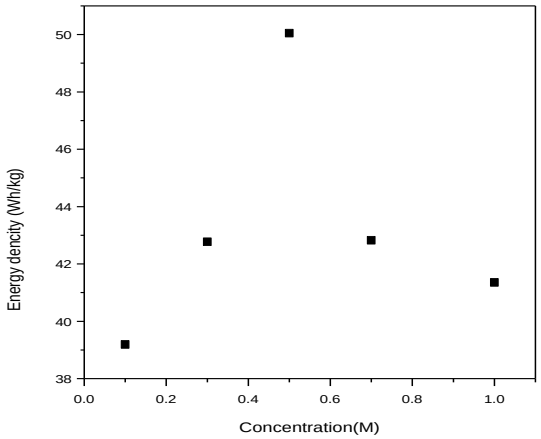

Fig. 5: the concentration dependence of energy density in tested PS MnO_2/SS electrode in different concentrations Na_2SO_4 electrolytes.

From this figure it is clear that the value of max energy density equals 50.045 Wh/kg and corresponds to concentration of 0.5 M Na_2SO_4 electrolyte. This means that the most suitable Na_2SO_4 electrolyte concentration used electrolyte solution to construct MnO_2 based supercapacitor of maximum specific capacity 341.99 F/g is 0.5 molar. This may be due to that, the increase in Na_2SO_4 concentration from 0.1 M to 0.5 M meets the increase of the electric polarization due to high degree of freedom of the molecular orientation under the effect of the resulted electric field.

On the other hand, the increase of the Na_2SO_4 concentration more than the above critical value (0.5 M) may cause the start to reduce the freedom of the molecular orientation under the effect of the resulted electric field of the decreased electric polarization. This decrease in the molecular orientation mobility increases as the electrolyte concentration increases than 0.5 M. This leads to the observed decrease in the polarization and thus the decrease of the specific capacity.

3.3 Charge-Discharge curves dependence on electrolyte concentration

Charge-discharge curves of the prepared electrodes were conducted using chronopotentiometry at current density 1mA/cm^2, for each concentration of Na_2SO_4 electrolyte and are shown in Fig. 6.

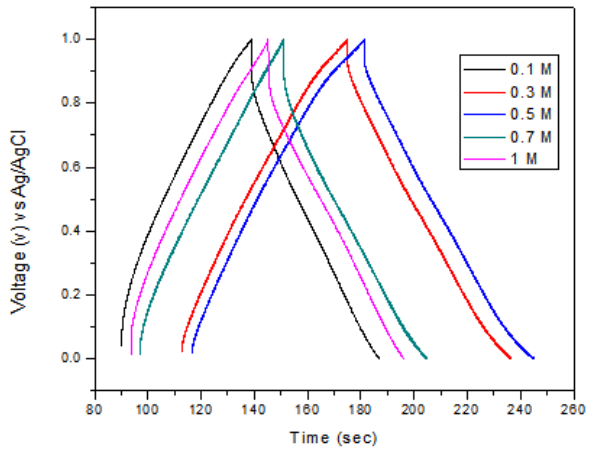

Fig. 6: Charge- discharge curves of deposited MnO_2 films at constant current density value 1mA/cm^2 for different concentrations of Na_2SO_4 electrolyte.

From the Fig. 6, it can be noticed, for each tested concentration, the voltage increases to reach maximum value at the end of the charging process half cycle. As, the voltage reaches max value, it starts to decrease during the other discharging half cycle. The values of specific capacitance were obtained from charge-discharge curves by using the following equation: SC = $\frac{I}{m}\frac{dt}{dv}$, where I is the discharge current, m is the mass of the MnO_2 film, and dv/dt is the slope of the discharge half cycle. Also, the time required for both charging and discharging, as well as, the time constants were all calculated for each electrolyte concentration and the obtained results are listed in Table (5).

Table (5): Specific capacitance, charging time, discharging time and time constant values, for tested PS deposited MnO_2 films for different electrolyte concentrations.

Concentration (molar)	Charging half cycle		Dis-charging half cycle		Specific Capacitance(F/g)
	Time (sec)	Time constant (sec)	Time (sec)	Time constant (sec)	
0.1	40	20	58	40	292.57
0.3	60	32	70	40	336.24
0.5	55	33	78	55	341.99
0.7	55	30	59	34	318.47
1	48	20	58	40	307.31

From the obtained results of Fig. 6 and table (5) we can conclude that the best concentration of Na_2SO_4 as an electrolyte solution to construct MnO_2 based super-capacitor of maximum high capacity is 0.5 Molar. This may be due to that the effect of increase of Na_2SO_4 concentration from 0.1 M to 0.5 M meets as was interpreted and mentioned previously in sec. 3.2 [25].

3.4 Electrochemical Impedance Spectroscopy (EIS)

Fig. 7: shows the Nyquist plots (the imaginary and real parts of the electrical impedence, Z_{imm} versus Z_{real}) for the (PS) deposited MnO_2 films as tested in different concentrations (0.1, 0.3, 0.5, 0.7, 1) molar of Na_2SO_4 electrolyte. Measurements were carried out in the frequency range (10 mHz -100 kHz) at 10mV amplitude.

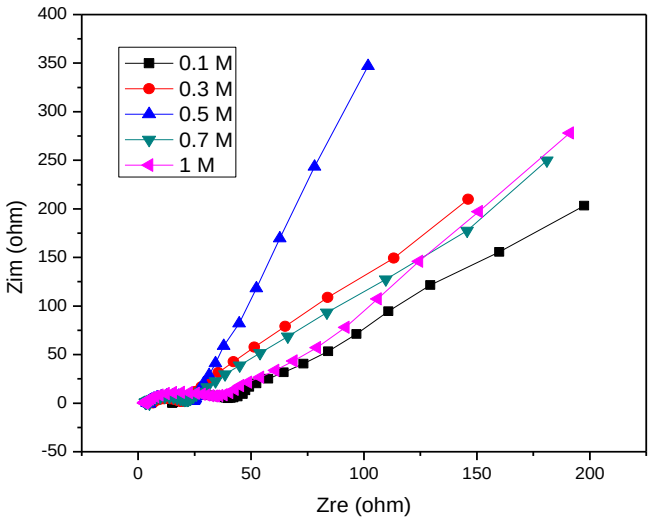

Fig. 7: Nyquist plots of MnO_2 deposited films investigated in different concentrations of Na_2SO_4 electrolyte in the frequency range of 10 mHz –100 kHz at 10 mV amplitude.

From this Fig. 7, it is obvious to note the dependence of Z_{im} on Z_{real} shows the appearance of semi-circles and a linear behavior at high frequency and low frequency regions, respectively. These results indicate the resistive and capacitive behaviors of the tested electrode. Intercepts of the obtained semi-circles at the beginning with the real impedance axis, gives the corresponding values of the equivalent series resistance (ESR) as a dominant resistive behavior of the constructed supercapacitor, while, values of the diameters of the obtained semicircles define the charge transfer resistance (R_{ct}) for tested electrode in each concentration [13, 30]. However, the values of both, ESR and R_{ct} were calculated and the obtained results are listed in Table (6).

Table (6): Values of R_{ESR}& R_{ct} for tested PS deposited MnO_2 films for different electrolyte concentrations.

Concentration(Molar)	R_{ESR} (Ω)	R_{ct} (Ω)
0.1	15.10	34.5
0.3	6.53	21.4
0.5	4.9	15.5
0.7	4.95	21.95
1	6.66	41.62

From Table (6) it is seen that the lowest values of both resistances, R_{ESR}& R_{ct} are 4.9 Ω and 15.5 Ωcorresponding to the value of 0.5 M concentration in agreement with all previous results of this work and confirms that anodic potentiostatic electrodeposited amorphous MnO_2 film on SS substrate and that reported [26] on the effect of manganese acetate concentration in the range, 0.05 to 1M may help good for supercapacitor application.

IV. CONCLUSION

In the present study, anodic potentiostatic electrochemical deposited MnO_2 amorphous film onto stainless-steel substrate was prepared. Effects of concentration of Na_2SO_4 electrolyte solution in the range, from 0.1 to 1 M, on the capacitive behavior, charging/discharging characteristics and electrochemical impedance spectroscopy were investigated. The obtained results revealed that cyclic I-V curves indicate the existence of high capacitive behavior as a good response to the charge carrier's polarizability. The specific capacitance decreases, from 308.52 to 183.9 F/g as the voltage scan rate increases from 10 to 100 (mV/s) and the highest maximum and average power obtained are 15 and 4.25 watt which corresponds to 0.5 M electrolyte concentration. Also, the max energy density =50.045 Wh/kg and corresponds to concentration of 0.5 M Na_2SO_4 electrolyte.

Investigation of charging/discharging processes, has led to the calculation of their time, time constants and specific capacitance and the results obtained revealed that the best concentration of Na_2SO_4 among all tested electrolyte solutions to construct MnO_2 based supercapacitor of maximum high capacity is 0.5 Molar. This was interpreted due to the different effect of Na_2SO_4 concentration below the 0.5 M and beyond which meets the increase as well the decrease of electric polarization according to freedom of molecular orientations,

Electrochemical impedance spectra of tested films in the range of 10 mHz to 100 kHz showed both the two resistive and capacitive behaviors which are dependent on the electrolyte concentration noting that lower values of both electrical series and charge transfer resistances are found as 4.9 Ω and 21.4 Ω, associated to the 0.5 M concentration of the Na_2SO_4 of electrolyte solution.

Finally, we can conclude that the best concentration of Na_2SO_4 as an electrolyte solution to construct MnO_2 based supercapacitor of maximum high capacity is 0.5 Molar.

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