



Sustainable sol–gel synthesis of NiMn_2O_4 /activated carbon/PVDF nanocomposites for energy storage devices

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Nickel–manganese oxides (NMO) prepared using the Sol-Gel process for use in energy systems as cathode in Li- ion batteries. Structural properties are studied for NMO powders using XRD. The findings suggest that a crystalline cubic structure is displayed during the production of NMO. Atomic Force Microscopy (AFM) indicate that the average diameter is 39.73 nm. Scanning Electron Microscopy (SEM) for NMO shows uniform spherical structures. The thermal analysis using thermogravimetric analysis (TGA), revealed that NMO is thermally stable, absorption measurement for oxide with different percentages of PVDF and activated carbon are measured using different pH, acidic solution exhibits higher absorption by nanocomposites.

Keywords: Sustainable; Sol–Gel; Nanocomposites; NiMn_2O_4 ; Energy storage devices.

1. INTRODUCTION

Metal oxides are fundamental inorganic compounds that have long been explored for their diverse applications in nanotechnology across various scientific and technological domains. Recently, their

unique electronic, chemical, and structural properties have positioned them as a crucial class of materials in the development of advanced energy storage systems [1-3].

Specifically, Nickel-Manganese Oxides are of particular interest as cathode materials for Lithium-ion Batteries (LIBs) due to their high theoretical capacity and structural stability. The synthesis and characterization of these materials are paramount to optimizing their performance. The Sol-Gel process, a cost-effective and sustainable method, is often employed to prepare these oxides, allowing for precise control over stoichiometry and nanostructure [4-6].

Current research efforts are heavily focused on tailoring the material's properties through various modifications and composite formations. For instance, studies have investigated the structural and magnetic properties of metal oxides and ferrites is a key strategy to create high-performance nanocomposites that enhance conductivity, mechanical stability, and overall cell capacity. Research Focus on Related Materials includes significant work on related compounds and nanocomposites [7-10].

2. EXPERIMENTAL

2.1. Preparation of NiMn_2O_4

"Sol-gel process" is employed as sustainable method for producing metal oxides using (10) g of precursor salt- fuel acid is weighed on a single pan balance, dissolved in the necessary volume of deionized water, and vigorously mixed. Three distinct beakers are filled to stoichiometric levels with acetates-based Nickel, Manganese and chelating agent in a ratio of 1:1:2 with pH (5.2). The obtained standardized solution is thoroughly stirred in a magnetic stirrer at (80–90) °C for four to six hours, or until gel formation, in order to remove the excess water. The gel-filled beaker is first cooked in a hot air oven for 12 hours at 150°C. Powder is then heated in an oven for 6 hours at 700 °C, Figure (1).



Figure 1 Powder of NiMn_2O_4 Calcined at 700 °C.

3. RESULTS AND DISCUSSION

The pattern XRD of Nickel–manganese oxides NiMn_2O_4 reveals the formation of the NiMn_2O_4 cubic structure, Figure (2) that correspond to the $C12/m1(12)$ symmetry $Fd-3m$ (227) with a lattice parameter $a = 8.40280\text{\AA}$, agree with powder of NiMn_2O_4 obtained from the International Center of Diffraction Data Card (JCPDS 96-591-0162) and in the literature ($a = 8.3790\text{\AA}$, ICDD card 88-0241) and ($a = 8.40\text{\AA}$, ICDD card 71-0852).

The main peaks for planes (222), (400), (331), (422), and (440) at the values of 2θ (35.39°), (43.40°), (47.14°), (53.92°), and (62.68°) are investigated. For the significant peak (422), the crystallite size, dislocation density, and strain of the sample shows to be 7.7 nm , 16.77×10^{15} , and 0.0045 respectively. The dislocation density increases with the decrease in crystalline size [11,12] as displayed in Table 1.

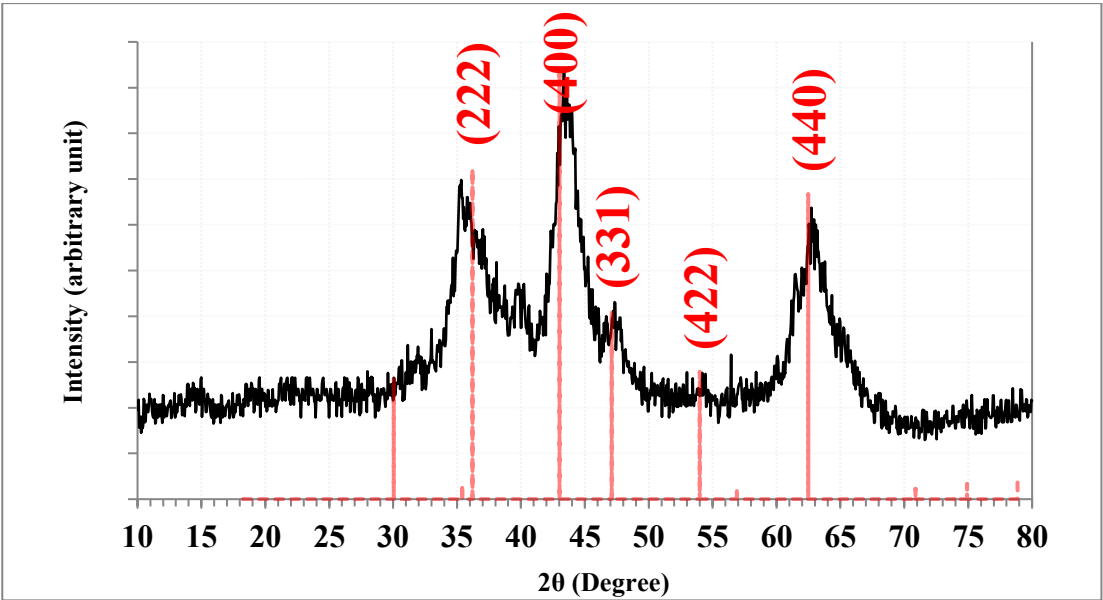


Figure 2 XRD spectra of NiMn_2O_4 at $700\text{ }^\circ\text{C}$.

Table 1 The XRD properties of NiMn_2O_4 powder.

2θ (Deg.)	FWHM (Deg.)	d_{hkl} Exp.($\text{\AA}$)	C.S (nm)	hkl	$\delta \times 10^{15}$ (line/ m^2)	ϵ
35.3928	2.5121	2.5341	3.3	(222)	90.75	0.0104

43.4045	2.1727	2.0831	3.9	(400)	64.57	0.0088
47.1387	1.3579	1.9264	6.4	(331)	24.55	0.0054
53.9282	1.1542	1.6988	7.7	(422)	16.77	0.0045
62.6867	3.0553	1.4809	3.0	(440)	107.89	0.0114

3.1. AFM of NiMn_2O_4 powder

The AFM pictures for the NiMn_2O_4 is shown in Figure (3). The image shows a uniform surface; indicate that the particle has uniform dimensions. The average diameter on the surface for NiMn_2O_4 is (39.73 nm).

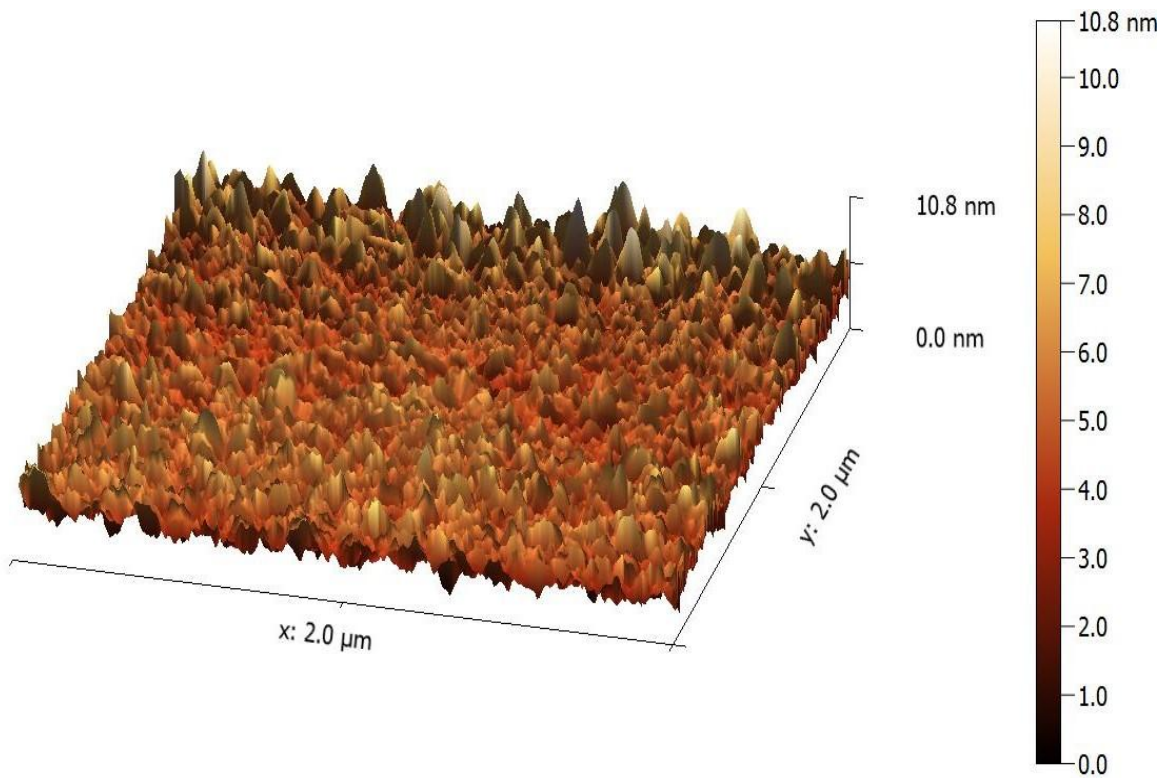


Figure 3 AFM images for the NiMn_2O_4

SEM images for NMO at different magnifications is shown in (Figure 4). The surface morphology of NMO is characterized by particles distribution at different size with grain boundaries of uniform distribution, the micrographs of the NMO synthesized samples shows also a porous structure and nearly rod-shaped grains with some agglomeration [13,14].

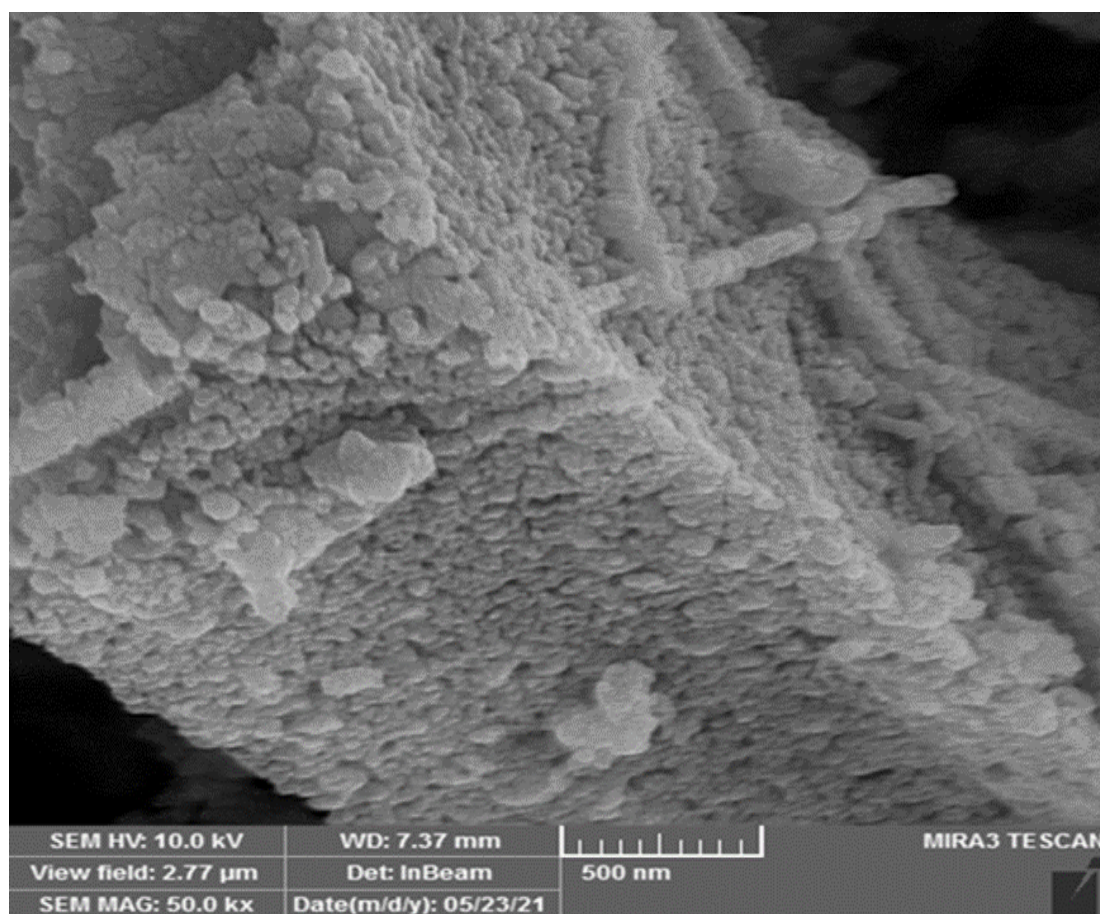


Figure 4 SEM for NiMn₂O₄ powder.

TGA data indicate mass loss close to 100 °C, Figure5, corresponding to the evaporation of water, and another one near 310 °C, attributed to the decomposition of the organic residuals related to the precursor addition. In this process, occurring up to 600 °C, the mass loss can mainly occur due to the formation of the compound [15-18].

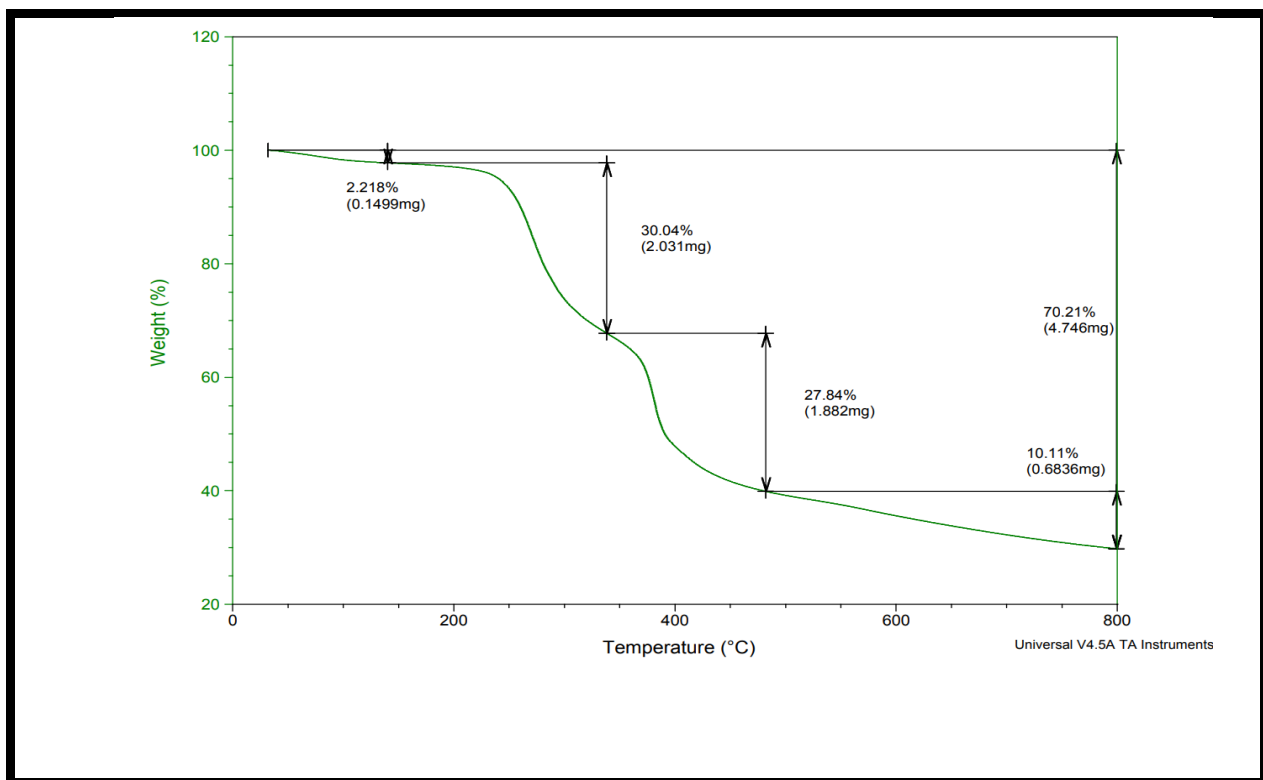


Figure 5 Thermo-gravimetric curve of NiMn_2O_4 .

3.2. pH-dependent absorption for NiMn_2O_4 nanocomposites

Water absorption is defined as the amount of water absorbed by a material and is important to understand the performance of the materials to access the type of the required electrode through processing depending on the filler types and PH. The presence of moisture in the material structure also influences thermal insulation and dielectric properties. Some of the known effects of water

absorption include dimensional, and mass changes caused by water absorption, extraction of water-soluble components, and changes in mechanical (elasticity, tensile strength, impact strength) and electrical performance.

In addition, the selection of any material, for a specific application, cannot be made without finding out the effect of the environment on its characteristics. The amount of water absorbed is related to the porosity and the amount of available liquid water at the surface of the material. For this reason, the porous material can uptake and store more water whereas the nonporous (dense) material can store a limited amount of water also the rate of diffusion increased with decreasing density of atomic packing. In this test, the nanocomposite materials are immersed in three media of neutral (PH=7), acidic (pH=3), and base (pH=11) for 150 days to study the percentage of absorptivity of such solutions in these materials and to compare the hygroscopicity the water absorption is calculated by measuring the weight of the sample before and after hydration [12,13]. The immersing of the samples in solutions leads to the release and removal of any residual monomer the solute diffuses into and is absorbed within the polymer, the small solute molecules fit into and occupy positions among the polymer molecules. Thus, the macromolecules are forced apart such that the specimen expands or swells. Furthermore, this increase in chain separation results in a reduction of the secondary intermolecular bonding forces. This test is very important for studying the behavior of these materials when exposed to humid, acidic, and alkaline environments.

The effects of solution weight gain (neutral, acid, and base) on samples at various PVDF and activated carbon concentrations are illustrated in the figures (6), (7) and (8) shown below, the largest percentage of weight is for the acidic solution. Acidification of water helps in the dissociation of water into ions. Acids easily dissociate into ions in aqueous solution. It is found that activated carbon inclusion results, overall, in a significant drop of the total porosity so that diffusion of Nano modified specimens are lower. Based on microstructural analysis, it is suggested that activated carbon contribute to the refinement of the pore network as well as bridging of micro-cracks [19-22].

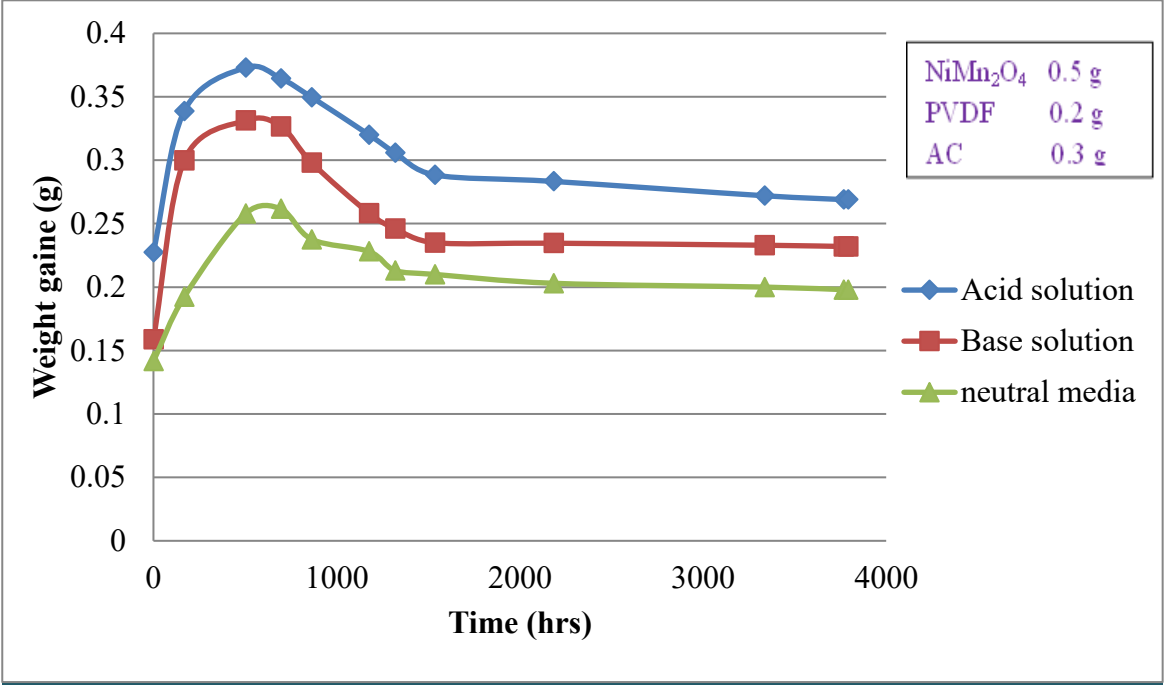


Figure 6 Weight gain variation with time at ratio 20% PVDF and 30% Activated Carbon (Ac) added to NMO.

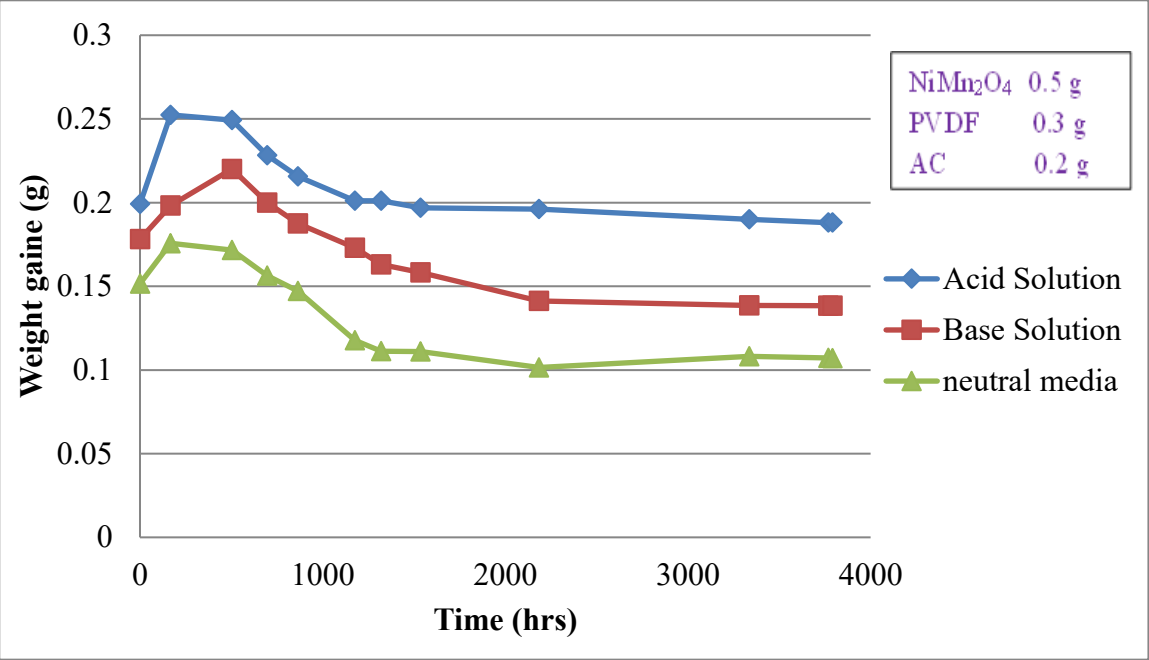


Figure 7 Weight gain variation with time at ratio 30% PVDF and 20% Activated Carbon (AC) added to NMO.

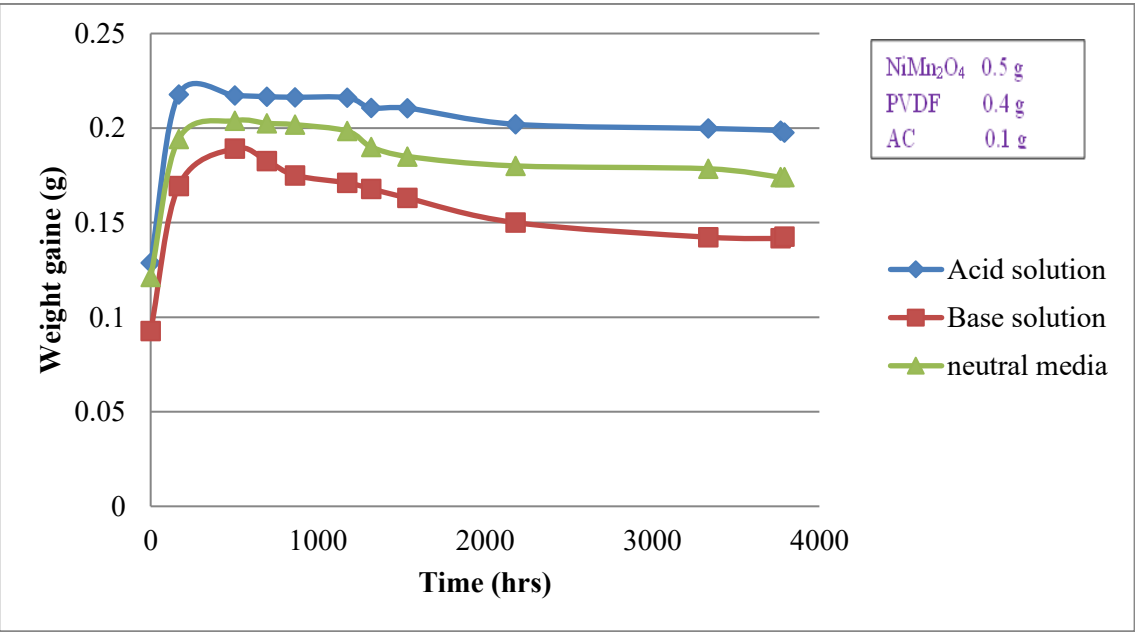


Figure 8 Weight gain variation with time at ratio 40% PVDF and 10% Activated Carbon (Ac) added to NMO.

4. CONCLUSIONS

NiMn₂O₄ nanoparticles were successfully synthesized via the sol–gel method and characterized using XRD, AFM, SEM, and TGA. The results suggest the formation of a crystalline cubic structure with nanometric particle size, uniform morphology, and good thermal stability. Incorporating PVDF and activated carbon into NiMn₂O₄ significantly influenced the porosity and water absorption behavior under neutral, acidic, and alkaline conditions, with the best performance observed in acidic media.

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