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Original article

Wet chemical synthesis and characterization of FeVO₄ nanoparticles for super capacitor as energy storage device



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ABSTRACT

Vanadates of transition metal found its potential applications in the fields of lithium ion batteries, gas sensors, photo catalysts, solar cells and so on. Among the metal vanadates, Iron vanadate is vital as an organic pollutant remedying, gas sensor material, and selective catalytic reduction material. This current research focuses on synthesizing iron vanadate nanoparticles by wet chemical synthesis with controlled pH using ammonia solution. The nanostructured FeVO₄ particle were characterized structurally with the powder XRD studies. Strong crystal planes were formed at Miller indices (111), (0–12), and (–220) which is confirmed from the intensity of the diffraction peaks. FT- IR and micro Raman studies was taken to identify the molecular vibrations present in the material and it shows that the sharpest apex at 508 cm⁻¹ obtained is attributed to the stretching oscillations of Fe–O and V–O–V modes. The Raman spectrum confirmed the separation of Fe – O, V – O, and different stretching fashions of V – O – Fe. The V–O stretching mode increases the intense bands showing the very high strongest peak of FeVO₄ because of electro negativity of iron the metal. The morphology of iron vanadate was confirmed with SEM analysis showing that particles are much isolated and cubical with few polyhedron structures. The electrochemical response of FeVO₄ evaluated the specific capacitance at 10 mV/s as 402 Fg⁻¹ Energy density values were calculated as 16.08 Whkg⁻¹, 13.08 Whkg⁻¹, 10.2 Whkg⁻¹, 8.76 Whkg⁻¹, 7.64 Whkg⁻¹ and 6.92 Whkg⁻¹ from the cyclic voltammetry profile at the slow rates varying from 10 – 100 mV/s. The magnetic properties were analyzed by measuring the magnetic susceptibility and magnetization using a VSM magnetometer and the results evident that the material exhibits the paramagnetic behavior.

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1. Introduction

Recently, energy storage devices such as supercapacitors, fuel cells and Li-ion batteries have gained much attention among researchers owing to the exponential growth of global population as a consequence of major energy crisis. Due to their appealing characteristics like high power density, long cycle life, etc., supercapacitors are considered as one of the most recent developments in the field of energy storage and conversion devices (Nithya et al., 2011; Deng et al., (2008); Mangamma et al., 1996). On the other hand, the energy density of supercapacitors is still lower than batteries, which limits their practical applications (Vuk et al., 2002; He et al., (2008)). Hence, proper selection of electrode materials is

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important to counter balance the charges and to obtain the finer electrochemical performance. Until now, plenty of orthovanadates (BiVO_4 , CeVO_4 , ZnVO_4 , MnVO_4 , and CuVO_4) were synthesized and employed as electrode materials for supercapacitor applications. Metal oxide nanoparticles were also utilized as electrodes to enhance the super capacitive performance. Sethuraman et al. have prepared $\text{Fe}_2\text{O}_3/\text{C}$ nanocomposites based negative electrode which delivers a specific capacitance of 315F/g in 2 M KOH electrolyte in the potential range of 0.2 to -0.7 V at 2 mV s^{-1} (Sethuraman et al., 2014). Similarly, Ghulam Mustafa and co-workers have prepared $\text{Mn}_2\text{O}_3/\text{Graphene}$ based composite materials exhibited a specific capacitance of 391F/g at a scan rate of 5 mV/s (Mustafa et al., 2021). Conversely, Mn_3O_4 was employed as a positive electrode which delivered the specific capacitance of 232.5 F g^{-1} at 0.5 A g^{-1} in the potential range of 0 to $+1.0\text{ V}$ and MnO_2 provided a specific capacitance of 411.9 F g^{-1} at 0.25 A g^{-1} in the potential range of -0.2 to $+0.8\text{ V}$ (Nithya et al., 2015). To the best of our knowledge, FeVO_4 is a promising material to resolve all these electrochemical issues. Vanadium exists stable in oxidation states varying from $+2$ to $+5$, and combines with various elements to produce several new compounds. FeVO_4 occurs in four types of polymorphs: $\text{FeVO}_4\text{-I}$, $\text{FeVO}_4\text{-II}$, $\text{FeVO}_4\text{-III}$ and $\text{FeVO}_4\text{-IV}$. Among these polymorphs, only $\text{FeVO}_4\text{-I}$ is stable at room temperature and remaining phases are not. They exist in metastable state at intense temperature and pressure. In $\text{FeVO}_4\text{-I}$, Fe^{3+} ions consists three crystallographic positions. Two of this positions are deformed octahedral FeO_6 and one site occurs in perverted triangular bipyramidal FeO_5 domains. The octahedral Fe-O forms six vertical lines of two fold hooked sequence. These sequences are

combined mutually by VO_4 triangular pyramids which in turn generate three dimensional sub-structures (Groń et al., 1991; Mishra et al., 2019; Quan et al., 2016). Previously, Aradhya Mishra et al. have utilized FeVO_4 and FeVO_4/rGO composites provided a specific capacitance value of 97.54F/g and 189.10F/g at 0.5 mA/cm^2 , respectively (Aladine et al., 2009). So far, FeVO_4 particles had been synthesized using several techniques such as, wet chemical route, flux method and hydrothermal method for magnetic, electrical and photocatalytic applications (Groń et al., 1991; Melghit and Mungi, 2007; Poizot et al., 2000; Sim et al., 2012; Kesavan et al., 2021). The objective of the present work is to synthesis FeVO_4 nanoparticles utilizing simple wet chemical co-precipitation route by modifying the pH of the suspension with the help of ammonia solution and to study its electrochemical and magnetic characteristics.

2. Experimental section

The FeVO_4 nanoparticles were synthesized by traditional wet chemical method using ferric nitrate $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, ammonium meta vanadate (NH_4VO_3) as the starting precursor. For the synthesis of FeVO_4 , 0.1 M of ferric nitrate and 0.1 M of ammonium meta vanadate was dissolved in de-ionized water individually under constant stirring. Ammonium meta vanadate solution was prepared at a temperature of $70\text{ }^\circ\text{C}$ as it has low solubility at room temperature. Ferric nitrate solution was mixed drop by drop into the ammonium meta vanadate mixture and swirled vigorously for 2 h . The pH of the suspension was sustained with the value of 3 by adding 0.01 M ammonia solution. To remove the organic residuals present in the precipitate, it was cleaned by using de-

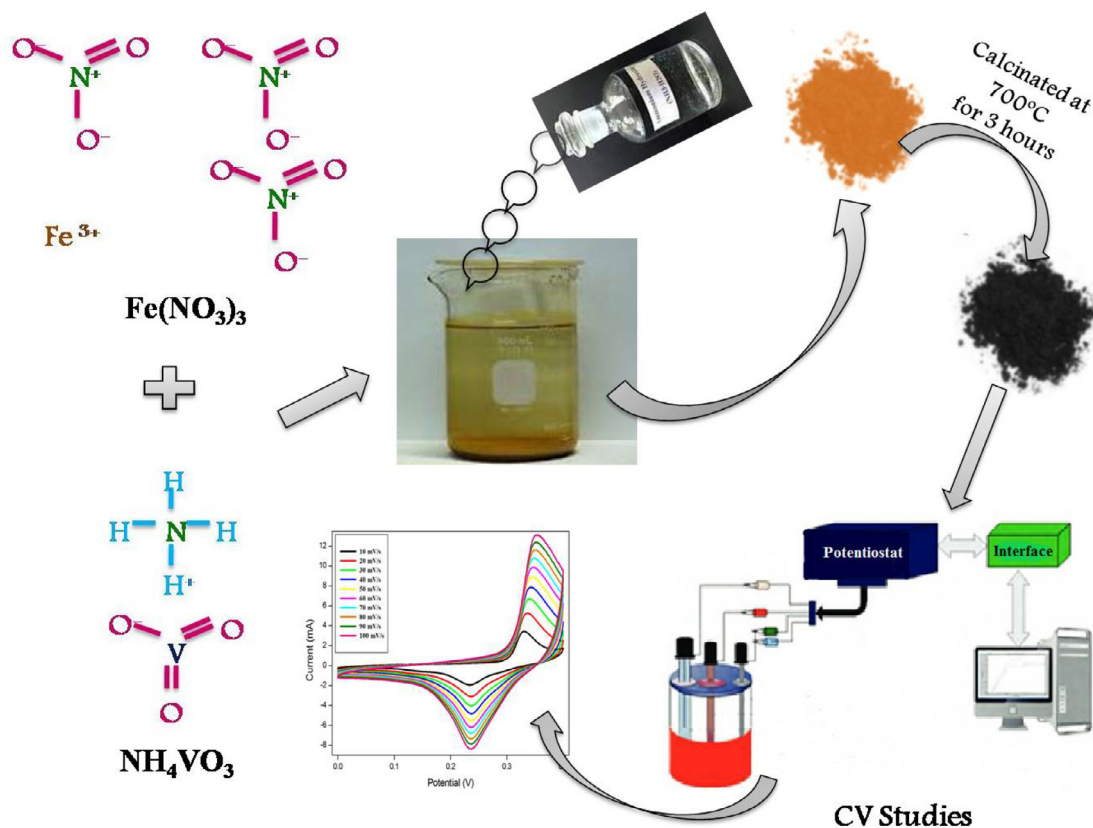
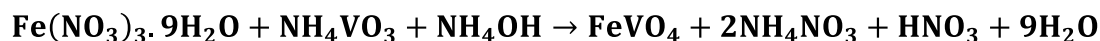


Fig. 1. Graphical representation for the synthesis scheme of FeVO_4 nanoparticles.

$$\delta = \frac{1}{D^2} \quad (3)$$

where λ is the wavelength of the X-ray source; θ is the Bragg's angle and β is the value of the full width half maximum.

The FeVO_4 nanoparticle was subjected to FT-IR analysis to identify the molecular vibrations in the form of nano-catalysts. Fig. 3 shows the peaks obtained for FeVO_4 nanoparticles were clear intensified bands, proposing the complete formation of appropriate chemical bonds (Nithya and Kalai Selvan, 2011). The apex at $3000\text{--}3500\text{ cm}^{-1}$ is ascribed to the O-H stretching vibration of the ingested water. The bending vibration of H_2O molecules is confirmed from the absorption peak at 1639 cm^{-1} . In addition, two more bands were observed, one is sharpest at 846 and the other at 929 cm^{-1} was consigned to an asymmetric stretching oscillation of V-O respectively. Mixed bridging of V-O-Fe stretching was observed from sharp peak at 673 cm^{-1} . The sharpest apex at 508 cm^{-1} obtained is attributed to the stretching oscillations of Fe-O and V-O-V modes, these results are similar to those reported by (Nguyen and Thanh, 2014; He et al., 2020).

Raman spectroscopy was used to prove the occurrence of crystalline nature. Raman spectrum performed at the normal temperature (Fig. 4). The well-defined peaks were obtained at $322, 369, 478, 731, 830, 897,$ and 920 cm^{-1} . The spectrum more evidently shows the crystallization property. The Raman spectrum confirmed the separation of Fe-O, V-O, and different stretching fashions of V-O-Fe. Zhang and his co-authors suggested that the peak at around 830 cm^{-1} was allocated to V-O stretching modes, and no splitting in the band represents the existence of degeneracy in the VO_4 tetrahedron symmetric stretching vibration.

The V-O stretching mode increases the intense bands showing the very high strongest peak of FeVO_4 because of the electro negativity of iron the metal. The significant peaks obtained at lower frequencies from 200 to 400 cm^{-1} are because of the V-O-V contortions and Fe-O stretching. The spanning V-O-Fe stretching oscillation was confirmed from the apexes at 731 and 830 cm^{-1} . The definitive apexes at 897 and 920 cm^{-1} are because of the stretching vibration of the terminal V-O stretching as reported by (Sethi et al., 2020; Yuan et al., 2012; Zhang et al., 2015). The spectrum of Raman bands of synthesized FeVO_4 shows many bands due to the triclinic structure of FeVO_4 , and non degenerate vibrations (Zhou and Li, 2012). The lower wave number bands were occurred due to the external modes of the lattice, vibrational and translational motions (Wang et al., 2022). From a crystalline point of view, all nanoparticles were compiled of V-O polyhedrons and some other metal-oxygen polyhedrons (Morton et al., 2010; Atta and Abdelhamied, 2021).

Fig. 5 represents the SEM micrograph of FeVO_4 nanoparticles obtained by calcinating at $700\text{ }^\circ\text{C}$. From the SEM image, it is evident that the particles are much isolated and cubical with few polyhedron structures. The SEM image contributes to the formation of regular polyhedron shape of FeVO_4 nanocatalysts (Zhu et al., 2014). At

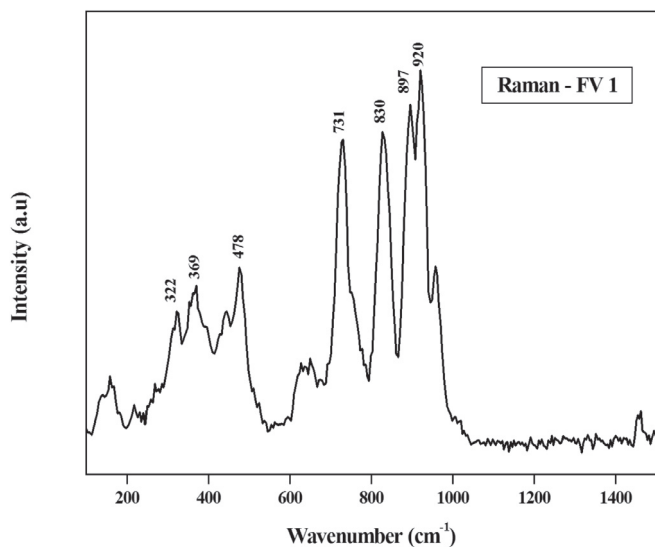


Fig. 4. Raman spectrum of the as-prepared FeVO_4 nanoparticles.

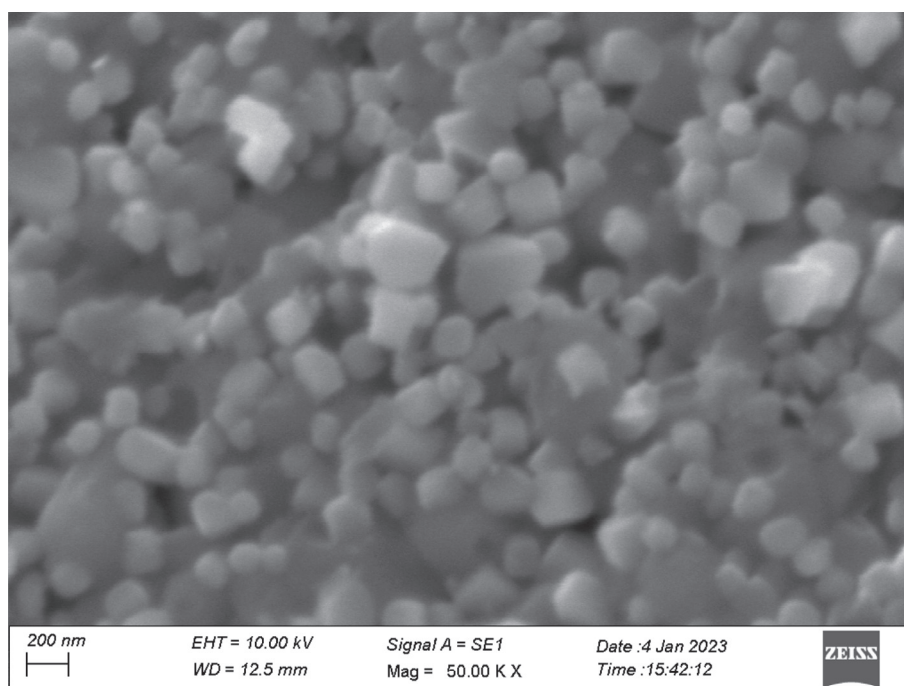


Fig. 5. SEM micrograph of as prepared FeVO_4 nanocatalysts.

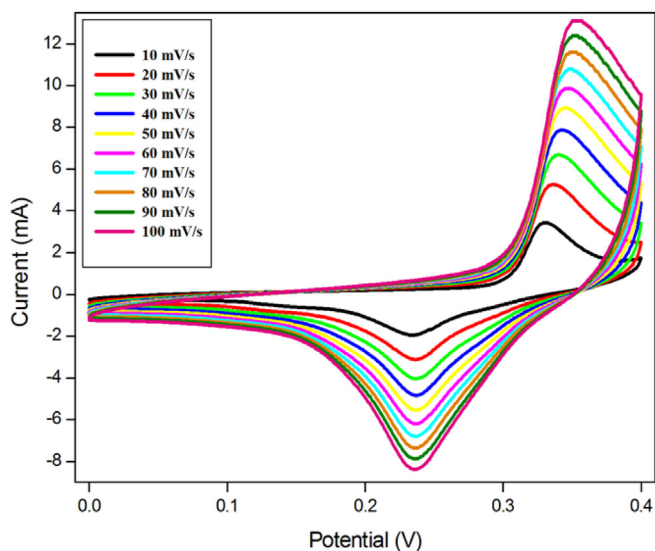


Fig. 6. CV curves for FeVO₄ super capacitor under KOH electrolyte.

some regions, the bigger nanoparticles are bounded by smaller nanoparticles.

Fig. 6 shows the cyclic voltammetry of the FeVO₄ nanoparticles under 2 M of KOH electrolyte. The cyclic voltammetry study is performed under slow rate varying from 10 to 100 mV/s. With increasing the scan rate, there is an increase in cyclic behaviour which is frequent in super capacitor behaviour. This is associated to the insufficient flow rate for the electrolyte ions to disperse in to the stomas of the nanoparticles (Ahmed Mohamed et al., 2020 Jul; Zhi Qiang He, 2020).

The cyclic curves have a good surface area which results in the great capacitance value and provide evidences of the electrochemical behavior of the supercapacitor (Nithya et al., 2015; Zhu et al., 2012). The specific capacitance is computed by the relation

$$C_p = \frac{\int IdV}{2km\Delta V}$$

Where $\int IdV$ is the integral area, k is the slow rate, m is the mass of the active substance and ΔV is the difference in the potential. The value of specific capacitance at 10, 20, 40, 60, 80 and

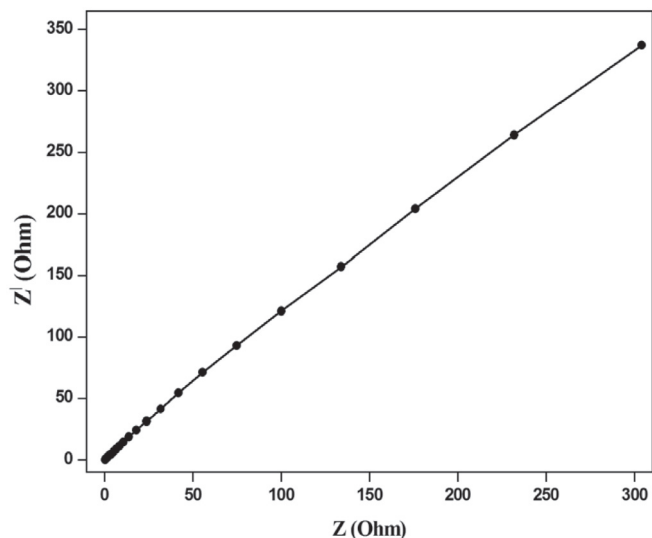


Fig. 7. Electrochemical Impedance Spectral curve for FeVO₄.

100 mV/s is 402 Fg⁻¹, 327 Fg⁻¹, 255 Fg⁻¹, 216 Fg⁻¹, 191 Fg⁻¹ and 173 Fg⁻¹ respectively. At lower scan rates, the specific capacitance is very high, whereas at higher scan rates it goes on decreasing.

Fig. 7 shows the electrochemical impedance nature of FeVO₄is recordedutilizing electrochemical impedance spectroscopy (EIS) in the frequency span from 10 mHz to 1 MHz. A very small and weak arc with a major single upright line was obtained at high frequency. The arc features to the charge transfer resistance (R_{CT}). Subsequently, the equivalent series resistance (R_s) is the meeting point of the impedance line on the real locus in the region of intense frequency. The series resistance is the mixture of three different resistances: natural resistance of the active substance, junction resistance at the active substance / current collector interface and ionic resistance of the electrolyte (Han et al., 2013; Li et al., 2014; Wang et al., 2016).

The energy density (E) is calculated from the following relation and its value depends on C_s, potential values V_{max} and V_{min}.

$$E = \frac{C_s (V_{max}^2 - V_{min}^2)}{2}$$

The energy density values are found to be 16.08 Whkg⁻¹ at the slow rate of 10 mV/s, 13.08 Whkg⁻¹ at 20 mV/s, 10.2 Whkg⁻¹ at 40 mV/s, 8.76 Whkg⁻¹ at 60 mV/s, 7.64 Whkg⁻¹ at 80 mV/s and 6.92 Whkg⁻¹ at 100 mV/s.

Fig. 8 clearly shows the specific capacitance of FeVO₄ nanoparticles is found to be 374.6, 237.5 and 134.9 Fg⁻¹ at 1, 3 and 5 Ag⁻¹ respectively. It is found that the specific capacitance is decreased with increasing the current density. The Comparison of electrochemical performance of prepared material with other reported materials as shown in Table 2.

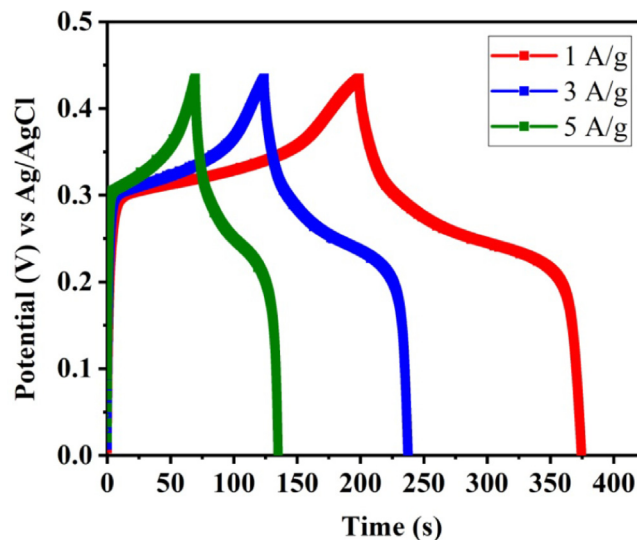


Fig. 8. Charge-discharge curve of FeVO₄ nanoparticles for different A/g values.

Table 2 Comparison of electrochemical performance of prepared material with other reported materials.

Material	Electrolyte	Specific capacitance (F/g)	Ref.
Mn3O4	1 M of KOH	355	31
Ni-Mn3O4	1 M Na2SO4	230	32
Graphene/Mn3O4	1 M Na2SO4	147	33
NiFe2O4	2 M KOH	368	34
FeVO4	2 M KOH	374.6	Present work

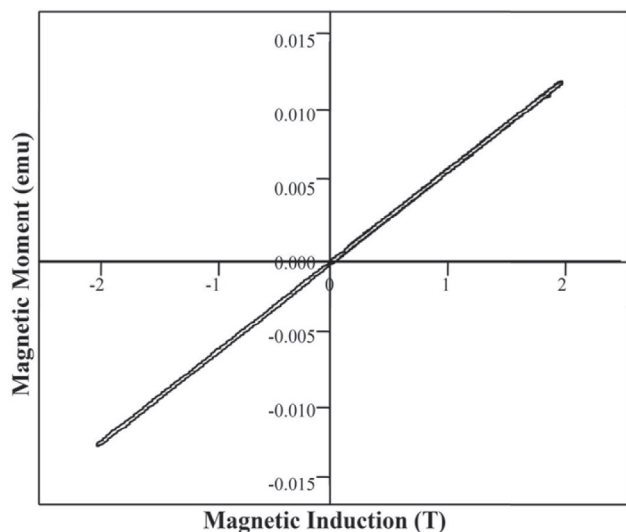


Fig. 9. Magnetic curve for FeVO₄ shows the plot of magnetic induction (T) vs magnetic moment (μ) for the iron vanadate.

The investigation into the magnetic properties of FeVO₄ was carried out with the purpose of gaining a better comprehension of the influence that the surfactant has on the magnetic properties of the material. The VSM magnetic measurements for the as prepared FeVO₄ NPs in Fig. 9 show the magnetic properties of resulting nanoparticles. Magnetization is the degree to which an applied magnetic field aligns magnetic dipoles inside a material. Samples exhibit a linear relationship, and the magnetic moments are not saturated up to an applied field of 50 kOe. It's possible that the samples' well defined magnetic ordering is responsible for the observed linear behavior. This might be because of the surfactant's role in producing a magnetic structure that is properly aligned. In the case of transition metal oxides paramagnetic behavior exists at high temperatures and exhibits ordered magnetic structures at low temperatures. Antiferromagnetic materials have the spins in the antiparallel direction so that the net magnetic moment is zero (Nithya et al., 2011). The FeVO₄ nanoparticles exhibit paramagnetic behaviour at room temperature, with a saturation magnetization of 0.0015 emu g⁻¹. From the graph, it is evident that the material exhibits the paramagnetic behaviour.

5. Conclusion

Metal oxide nanoparticles were also utilized as electrodes to enhance the super capacitive performance. This study examined the potential work of FeVO₄ nano-catalysts were successfully synthesized employing wet chemical process with a controlled pH value using ammonia solution. Thus, it has advantage of producing high-purity materials with favorable stoichiometry, a very homogenous product, and enhanced reactivity at lower temperatures, all without the need for complex equipment. The material attains its major growth in the (012), (-201), and (1-12) planes which will be confirmed from the strong intensity obtained in that corresponding direction. The significant peaks obtained at lower frequencies from 200 to 400 cm⁻¹ are because of the V – O – V contortions and Fe – O stretching. The lower wave number bands were occurred due to the external modes of the lattice, vibrational and translational motions. The spectrum of Raman bands of synthesized FeVO₄ shows many bands due to the triclinic structure of FeVO₄, and non-degenerate vibrations. The SEM image contributes to the formation of regular polyhedron shape of FeVO₄-nano-catalysts. At some regions, the bigger nanoparticles are

bounded by smaller nanoparticles. In order to acquire cyclic voltammetry study, the FeVO₄ nanocatalyst was coated in nickel foam by doctor blade technique. The specific capacitance and energy density values are found to be 402 Fg⁻¹ and 16.08 Whkg⁻¹ at a scan rate of 10 mV/s, 327 Fg⁻¹ and 13.08 Whkg⁻¹ at 20 mV/s, 255 Fg⁻¹ and 10.2 Whkg⁻¹ at 40 mV/s, 216 Fg⁻¹ and 8.76 Whkg⁻¹ at 60 mV/s, 191 Fg⁻¹ and 7.64 Whkg⁻¹ at 80 mV/s, 173 Fg⁻¹ and 6.92 Whkg⁻¹ at 100 mV/s from cyclic voltammetry and electrochemical impedance spectral analysis. From the results obtained, one can say the controlled pH with ammonia solution has sufficiently increased the super capacitance value of FeVO₄ when compared with other reported literatures.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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