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Effect of Vanadium Nano-fibrous Flowers decorated on Carbon Nano Materials for Enhancing its Hydrogen Storage Capacity

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Abstract: ***To enhance hydrogen storage capacity of Carbon Nano Materials (CNMs), Transition metal Vanadium in its Nano form at different concentrations on CNMs were synthesized from natural waste, Sugar-Cane Bagasse (SCB). Raw Carbon obtained by pyrolyzing SCB at high temperature in an inert medium and activated by alkali were decorated with Vanadium Nano-fibrous Flowers (V-NFs) (The authors named this as 'Vanadium Nano-fibrous Flowers') by annealing at different temperatures in CO_2 atmosphere. XRD and Raman spectroscopic analysis were carried out for its structural elucidation. EDAX and ICP-AES gave the amount of Vanadium being deposited. Nano-fibrous Flowers of Vanadium on porous morphology of CNMs was depicted by SEM analysis. Comparative studies of Hydrogen adsorption of CNMs were concluded by Sievert's apparatus for various concentration of vanadium on CNMs.***

Keywords: Sugarcane Bagasse (SCB), Carbon Nano Material (CNM), Vanadium Nano-fibrous Flowers (V-NFs), Hydrogen Storage Capacity, Sievert's apparatus, Hydrogen Adsorption

I. INTRODUCTION

Depletion in fossil fuel and increasing demand for energy supply with the rising population urged scientist to pursue for alternate energy source that will quench the need of future requirement [1][2]. In addition, it should not affect the environment adversely and sustainable at a relatively low cost [3].

Hydrogen as a fuel will be considered in future Energy Economy as it is high in energy density, light weight and being eco-friendly. Hydrogen is found to be abundant on earth and proves to be a sustainable and promising alternative fuel, generating significant energy and water as a by-product. Hence, it is considered as a green energy [4][5]. Hydrogen is used in fuel cells [6], as a safe and convenient fuel source which can be generated from renewable source [7][8]. Cost for hydrogen storage is currently not competitive with hydrocarbon fuels on practical large scale [9]. Hydrogen store in its compressed form under pressure is being unsafe to use it as mobility fuel [10]

Adsorption phenomenon can be utilised for hydrogen storage to lessen its hazardous consequences by limiting its dispersion thereby making hydrogen gas safe to store. Various material showing good chemisorption and physisorption can serve as better hydrogen storage equipments wherein physisorption is best suited for reversal desorption to generate hydrogen when needed in less efforts. Micro-porous materials shows better hydrogen physisorption phenomenon hence can be utilised for better efficient storage material [11]. CNM being porous and having good mechanical strength serves best option for hydrogen storage. Further, the CNM cost is reduced in vastly when produced from renewable source thus that will lower the storage cost thus fulfilling Hydrogen criteria as fuel for future economics.

Hence CNMs from the renewable sources are explored for better and economic hydrogen storage [12]. US-DOE has proposed 6.5 wt% as hydrogen storage capacity of material to be commercialised for future energy driven technology [13].

In this work, Effect of V-NFs being decorated on the CNMs from plant waste, sugarcane bagasse (SCB) [14] on Hydrogen adsorption is studied for various amount of Vanadium on CNMs.

II. EXPERIMENTAL TECHNIQUE

A. Synthesis of V-NFs Decorated CNM

The SCB was pyrolyzed at high temperature, activated and decorated with V-NFs in carbon dioxide atmosphere to get highly porous CNMs, for different VNFs concentration which were labelled as S1 and S2 [15][16][17].

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B. Hydrogen Adsorption by Sievert's Apparatus

CNM samples of around 5g were loaded in a sample holder in furnace of Sievert's apparatus wherein adsorption of hydrogen was studied by using Van der Waals real gas equation at a pressure of 60 bars at ambient temperature [15][16][17][18]. The observations at different concentration are tabulated in below Table I.

TABLE I: HYDROGEN ADSORPTION OF CNMS

S. No.	CNM	Tapped Density	Hydrogen Adsorption at 60 bar H_2 pressure and ambient temperature
		g/cc	% w/w
1	S1	0.2050	3.81
2	S2	0.3080	2.30

III.RESULTS AND DISCUSSIONS

A. XRD Analysis of CNMs

X-Rays Diffraction (XRD) analysis was carried using Malvern Panalytical's Empyrean diffractometer having X-ray tube with Cu target of wavelength as Cu Kα-1.54184 Å and X-ray generator of 45kV & 40mA. XRD diffraction peaks were analysed in continuous scanning mode with a scanning range of 10–90° of Diffraction angle (2θ).

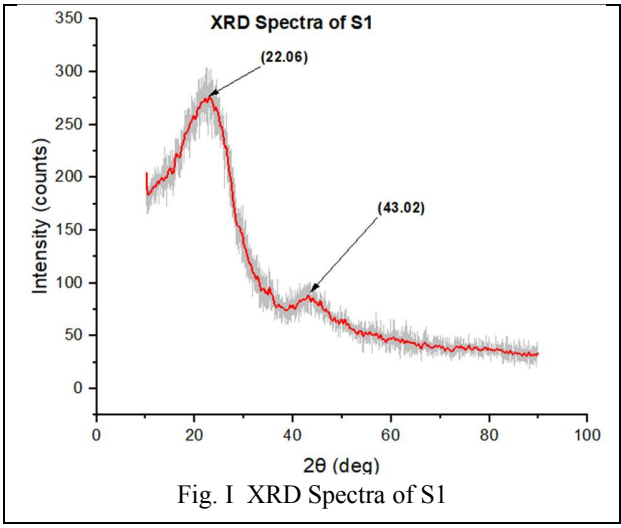


Fig. I XRD Spectra of S1

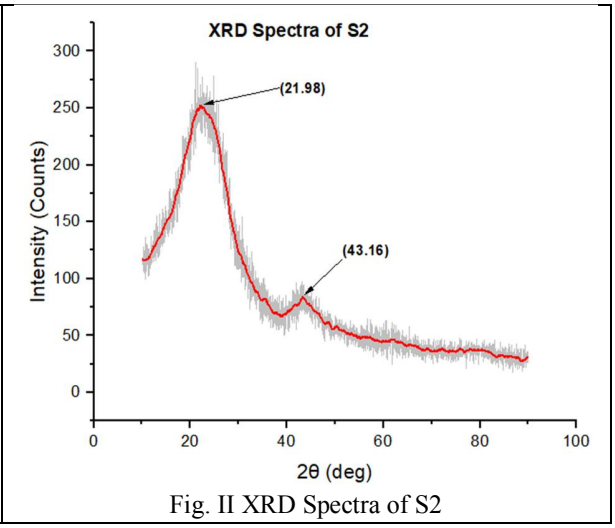


Fig. II XRD Spectra of S2

TABLE II PEAK TABLE OF XRD SPECTRA ANALYSIS OF CNMs

h,k,l Index	Standard 2θ in deg from ICDD card No. 00-056-0159 for Amorphous Carbon	S1	S2
		2θ in deg	2θ in deg
(0, 0, 2)	21	22.06	21.98
(1, 0, 2)	43	43.02	43.16

From the ICDD card No. 00-056-0159 for amorphous carbon and from XRD spectra in Fig. I and Fig. II and Table II and the broad peaks at 2θ = 22.06° and 21.98° diffraction angle in the spectra S1 & S2 spectra respectively of plane (0, 0, 2), indicate the presence of amorphous phase of reduced Graphene Oxide (rGO) with the confirmation peak at 43° of plane (1, 0, 2) being broad and week [18][19][20][21]. As there is no peak observed in spectra related to Vanadium oxides as mentioned in JCPDS 98-000-2338, ICSD-199 & ICSD-3403 [22][23], oxides of vanadium are absent since the material has been treated at high temperature in CO_2 reducing environment.

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B. SEM and EDAX Analysis of CNMs

Scanning Electron Microscopy (SEM) analysis illustrates fine morphology of the CNMs. FEI Quanta 200 FEG SEM were used for unfolding the "Nano-fibrous Flowers" morphology of vanadium on porous CNMs of S1 in Fig III while Energy Dispersive X-Ray (EDAX) Analysis conducted on same surface gave surface concentration of V-NPs around 0.8% with overall uncertainty of 11.74% as depicted in the spectra in Fig IV. Major elements Carbon being 94.8 % and Oxygen is 4.3 % as indicated in the EDAX spectra in Fig IV infers the formation of defects as rGO as confirmed in XRD [15][24].

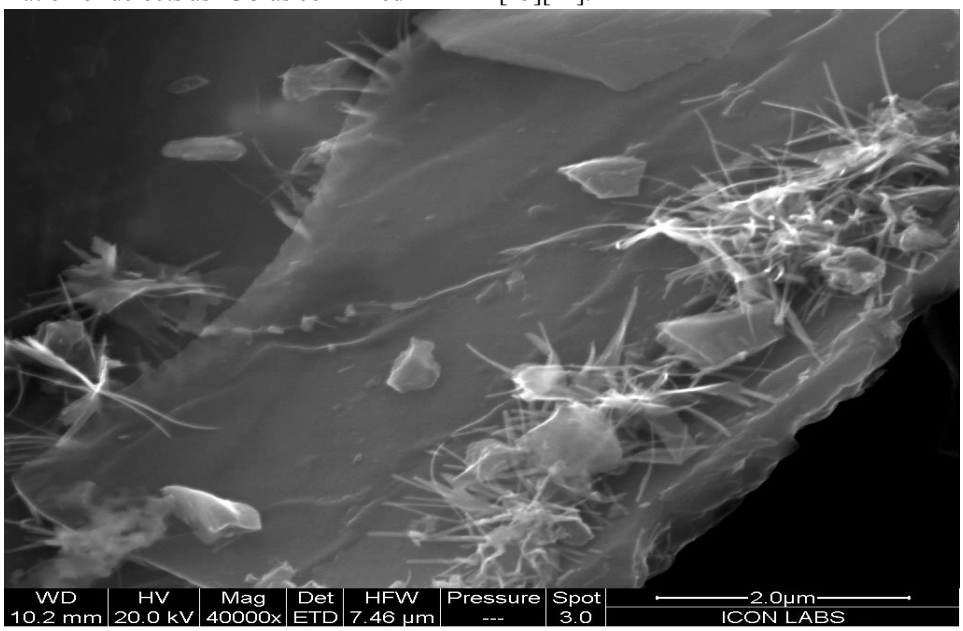
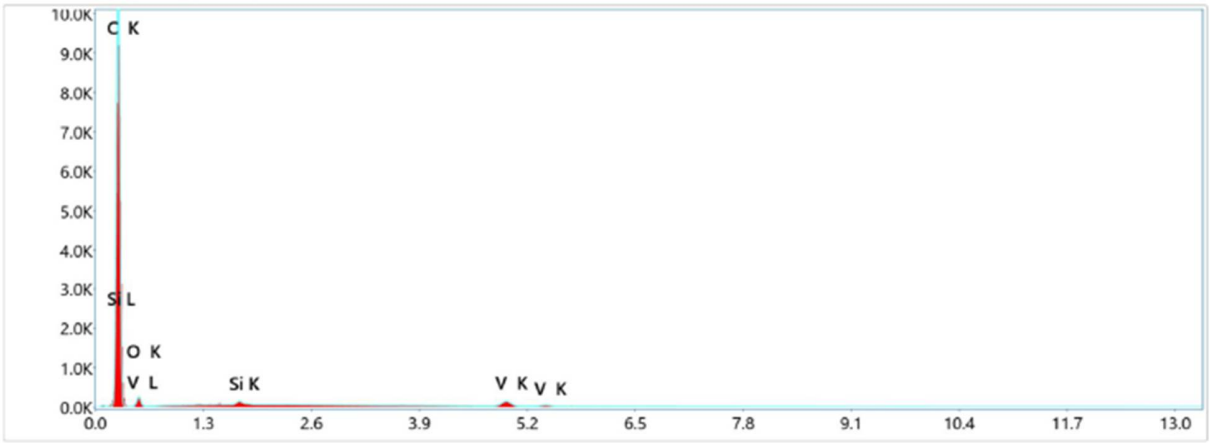


Fig III Vanadium Nano-fibrous flowers on CNM in S1



eZAF Quant Result - Analysis Uncertainty: 11.74 %

Element	Weight %	MDL	Atomic %	Net Int.	Error %	R	A	F
C K	94.8	0.04	96.5	1878.7	8.9	0.9427	0.2455	1.0000
O K	4.3	0.20	3.3	46.2	15.3	0.9499	0.0525	1.0000
Si K	0.2	0.03	0.1	27.1	14.1	0.9648	0.7700	1.0061
V K	0.8	0.11	0.2	53.1	8.2	0.9798	0.9906	1.0875

Fig IV EDAX Spectra of S1 with result table

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C. ICP-AES analysis of CNMs for VNF on CNM

Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) of CNMs was analysed for estimating the amount of VNF being deposited on CNMs. Accurately weighed 0.1g ± 0.001g of CNMs were digested for approximately 10 minutes in 10ml of 1:1 HCL in water of Millipore [15]. Analytical grade HCl reagent was used. It was then extracted with Millipore water which was filtered and then the filtrate further diluted in 100mL pure Millipore water. ARCOS ICP spectrometer was used for ICP-AES analysis whose RF generator of 1.6 kW, 27.12 MHz and wavelength range was of 130-770 nm with resolution of 9 picometer. The prepared solutions were aspirated in this ICP-AES and analysed results being tabulated in below Table III. Concentration of VNF's in sample S1by ICP-AES are matching with EDAX results of S1 as 0.8%w/w.

Table III VNF Concentration of CNMs by ICP-AES

Sample	w/w percentage of VNF in CNMs
S1	0.774
S2	0.945

D. Raman Spectroscopic Analysis of CNMs

Raman Spectroscopic Analysis of sample S1 and S2 were done by using Renishaw's Invia Reflex Raman Spectrometer having Laser HeNe as excitation source of of wavelength 633 nm with Power 17 mW which produces Raman wavenumber of Spectral Range 200nm–1600nm.

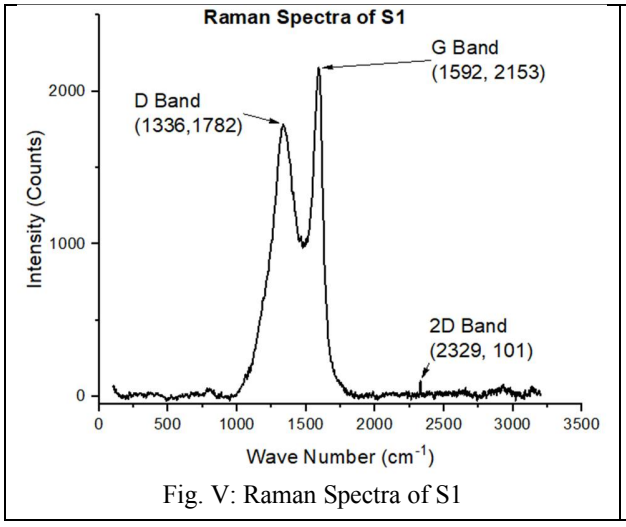


Fig. V: Raman Spectra of S1

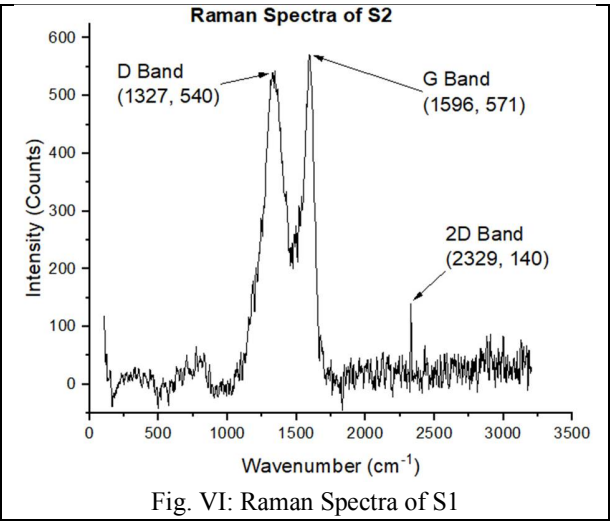


Fig. VI: Raman Spectra of S1

TABLE IV: PEAK TABLE OF RAMAN SPECTRA ANALYSIS OF S1 AND S2

Parameters	S1			S2		
	Peak-1 D-Band at 1336 cm^{-1}	Peak-2 G-Band at 1592 cm^{-1}	Ratio 0f D/G	Peak-1 D-Band at 1327 cm^{-1}	Peak-2 G-Band at 1596 cm^{-1}	Ratio 0f D/G
Intensity	1782	2153	0.83	540	571	0.95
Area	313977	385134	0.82	123916	132766	0.93

Raman analysis gave observation of two peaks at around 1336cm^{-1} and 1592cm^{-1} as D-band and G-band as shown in its spectra in above Fig. V and Fig. VI pertaining to Distorted and graphitic sp^2 carbon respectively whose intensity and area ratio calculated as 0.83 for S1 and 0.95 for S2 confirms that the distorted sp^2 graphene carbon increases with the increase in VNF's concentration during synthesis of CNMs from SCB [13]. Less intense Peak at 2329cm^{-1}in S1 & S2 spectra representing 2D band as an overtone of D band of distorted sp^2 graphitic carbon can be distinctly seen [24].

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IV. CONCLUSION

From all above observation, it is concluded that with the increase in VNF concentration, I_D/I_G ratio of Raman spectra increases, indicating the increase in the hetero atom of vanadium in graphitic carbon [24]. Hence greater the deposition of V-NFs on the CNM formed and greater graphitization as observed from the Raman spectra, the hydrogen adsorption capacity of CNMs decreases.

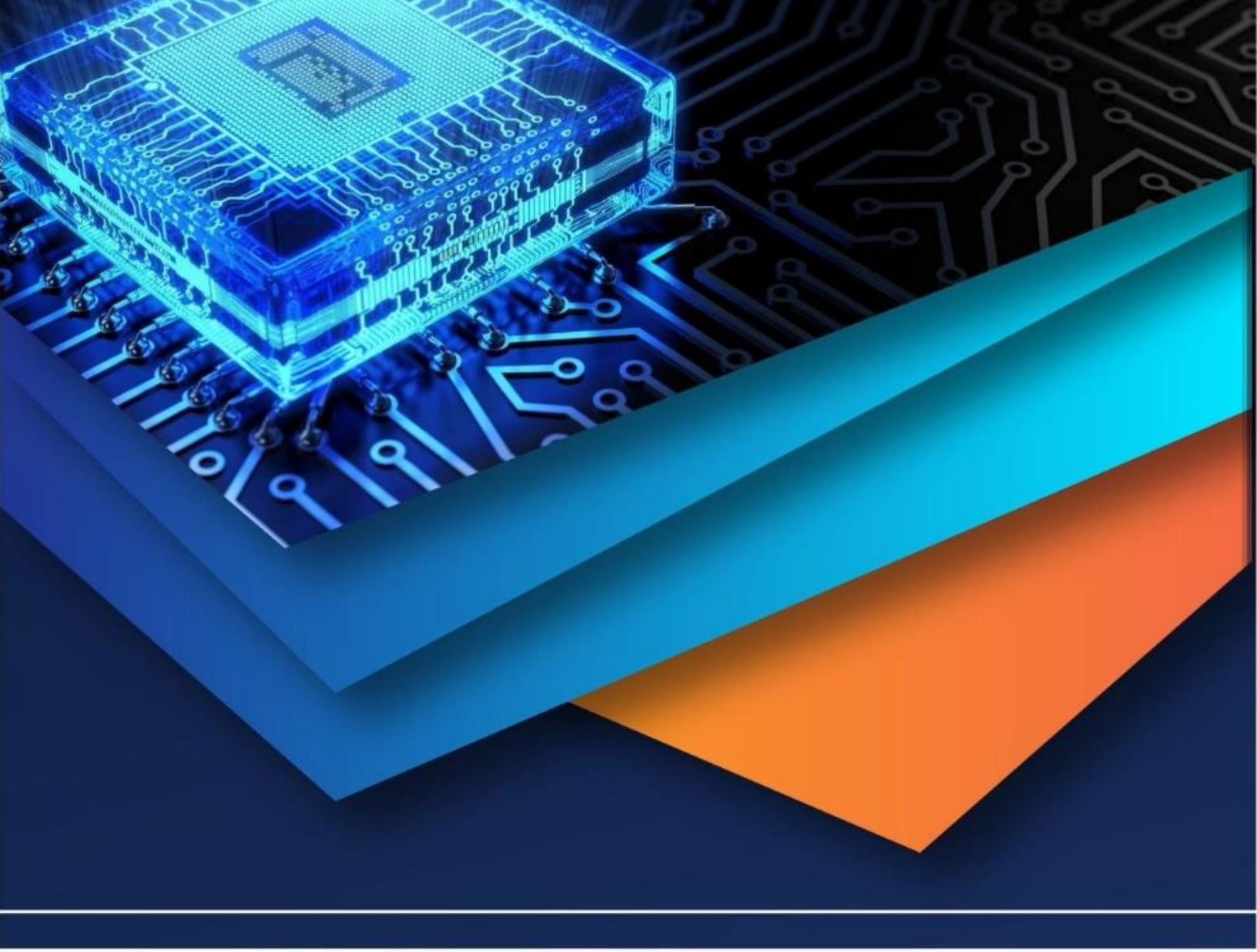
V. ACKNOWLEDGEMENT

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