iJRASET
International Journal For Research in
Applied Science and Engineering Technology

INTERNATIONAL JOURNAL FOR RESEARCH
IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 11 Issue: XII Month of publication: December 2023

DOI: https://doi.org/10.22214/ijraset.2023.57577

International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor: 7.538
Volume 11 Issue XII Dec 2023- Available at www.ijraset.com

Synthesis and Application of Bimetal Decorated Plant-Based Carbon Nanomaterial for Hydrogen Storage

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Abstract: In this study, carbon nanomaterials (CNMs) were synthesized from waste cotton fibers and examined their hydrogen adsorption capacity. The CNMs were created by subjecting waste cotton fibers to high-temperature pyrolysis at 800°C, followed by activation with alkali solution. Nano-sized metallic particles of Nickel and Aluminium were decorated on the activated carbon fibers. Analysis through SEM, EDAX, Raman spectroscopy, X-ray diffraction and BET surface area confirmed the porous nature and presence of metal particles in the CNMs. These materials exhibited a notably larger specific surface area (SSA) compared to untreated carbon fibers, enhancing their hydrogen storage capabilities as it showed 8.08 wt% of hydrogen adsorption using a Sievert's apparatus. This work, shows the promising potential of these CNMs for hydrogen adsorption in various industrial applications.
Keywords: Carbon nanomaterials (CNMs), Cotton Fibre, Bimetal, Hydrogen adsorption, Sievert's apparatus.

I. INTRODUCTION

The quest for sustainable and clean energy sources has gained significant momentum in response to the diminishing reserves of petroleum products and their contribution to environmental degradation [1]. D. J. Durbin and C. Malardier-Jugroot points out the recent advancements in hydrogen storage technologies and highlights the research areas where further work is needed to make hydrogen storage more efficient, safe, and economically viable for vehicles [2]. Hydrogen as a fuel offers efficiency, sustainability, and environmental friendliness, with the potential to replace non-renewable fossil fuels in various applications [3][4][5]. Hydrogen is being recognized as a promising alternative energy source, not only for automotive applications but also for powering smaller devices like laptops and smartphones [4].

Extensive research has been conducted to develop materials for hydrogen storage, including graphene-based materials, nanomaterials, complex hydrides, and many more [5][6][7][8]. Among the various techniques explored for hydrogen adsorption, physisorption on carbon materials has shown great promise due to its reversibility and lack of chemical bonding between hydrogen and the carbon surface [9][10][11][12]. Materials with weak Van der Waals interactions and porous structures are believed to meet the standards set by the U.S.

Department of Energy (DOE) for hydrogen adsorption at ambient conditions [13][14]. Researchers have sought to create carbon nanomaterials (CNMs) from plant-based materials to investigate their hydrogen storage capabilities [9]. Porous nanomaterials, such as carbon nanotubes, graphene, and carbon nanofibers, have garnered attention as efficient adsorbents for hydrogen due to their high specific surface area and porous nature, making them ideal substrates for enhancing hydrogen adsorption through metal enrichment [15]-[18]. Among the transition metals used in hydrogen adsorption, nickel stands out for its abundance, cost-effectiveness, and favourable adsorption properties [19]. Mukherjee et al. did a study aimed to maximize the hydrogen adsorption capacity of carbon fibres produced from cotton pyrolysis. This involved a series of steps, including pyrolyzing cotton in an argon atmosphere, loading those with metal nanoparticles at high temperature. The research utilized the Taguchi optimization technique to identify the most effective parameters for achieving high hydrogen adsorption [20]. Kailash et al. have shown advantages of bimetal decoration on CNMs over single metal for enhanced hydrogen storage [21]. Ultimately, the study successfully produced carbon fibres with a significantly enhanced hydrogen adsorption capacity.

This study aims to explore the impact of bimetal treatment (specifically, nickel in combination with other metals) on the hydrogen adsorption properties of CNMs derived from waste cotton fibres, offering insights into their potential for sustainable hydrogen storage solutions.

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II. EXPERIMENTAL TECHNIQUE

A. Synthesis of Carbon Nanomaterials (CNMs)

The cotton fibres derived from plant waste materials were pyrolyzed in a specially designed Lyndberg horizontal quartz furnace at 800°C in an inert atmosphere. Subsequently, these fibres were activated using (2N) NaOH solution and treated with nanoparticles of nickel and aluminium by using a mixture of solution of Nickel Nitrate (0.1N) and Aluminium Chloride (0.1N). This highly porous carbon nanomaterial was then employed to investigate its hydrogen adsorption properties, using Sievert's apparatus.

B. Measurements of Hydrogen Adsorption

A comprehensive investigation into hydrogen adsorption was undertaken, employing a standardized sample quantity of 5 grams in weight. The evaluation of adsorption capacity was carried out through the volumetric method at 50 bars, utilizing Sievert's apparatus under ambient temperature conditions. The detailed results of this study are systematically presented and tabulated in Table I. The experimental samples, crucial to this study, were securely positioned within the Sievert's apparatus, which had undergone a rigorous 24-hour leak test to ensure its integrity and reliability.

Table I

Metal Combination treated on CNM	BET surface area (m^2/gm)	Pore Volume (cc/gm)	Hydrogen Adsorption wt%
Ni + Al	392.781	0.2356	8.08

III. RESULTS AND DISCUSSIONS

A. Morphology of Metal Decorated CNMs

The scanning electron microscope (SEM) is a key tool in materials science, offering high-resolution imaging to study the detailed morphology of materials. It provides valuable insights into surface features, textures, and structures at a nanoscale level, aiding in material characterization and research. For this study, SEM results are given below, Fig 1(a) shows the nature of CNM synthesized which is in the form of Carbon Nano sheets while Fig 1(b) shows the decoration of Al and Ni particles on CNM, particle size can be observed as less than 100 nm.

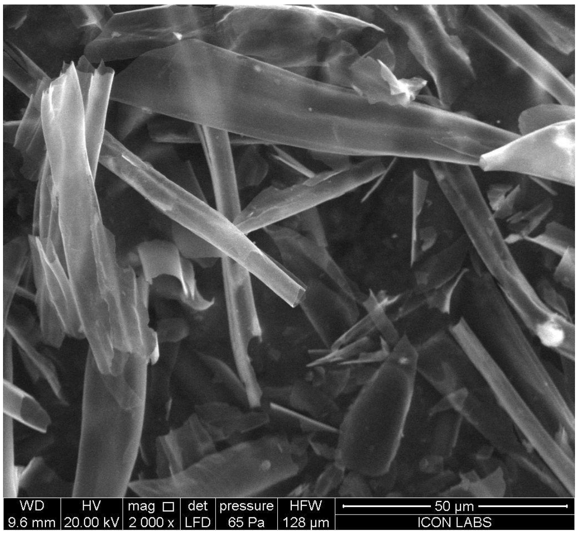

Fig. 1 (a). SEM image of obtained CNMs

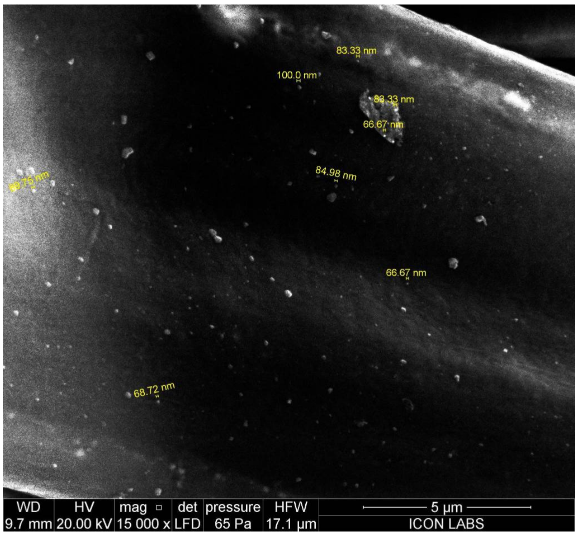

Fig. 1 (b). SEM image of CNMs decorated with Ni and Al nanoparticles

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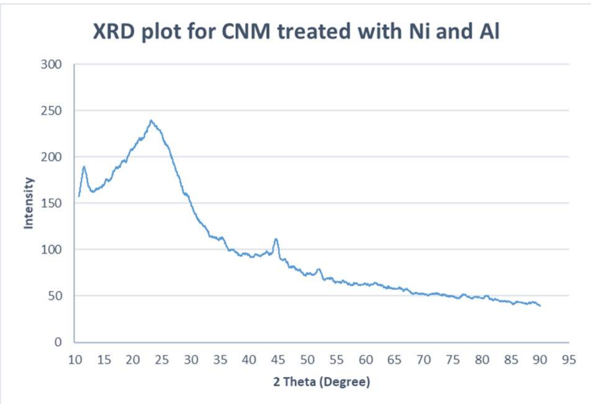
B. Characterisation of CNMs

1) X Ray Diffraction Analysis (XRD)

XRD technique is used to analyse the structure of materials. It involves exposing a sample to X-rays and measuring the diffracted X-rays at different angles. The resulting diffraction pattern allows to determine the crystalline phases, crystal orientation, and other structural information of the material Fig 2. shows XRD plot for CNMs decorated with Al and Ni nanoparticles in which peaks are observed at 44.42º and 55.17º of 2θ value which corresponds to [110] and [111] plane respectively (PDF card no. 9008802)

2) Raman Spectroscopy

Raman spectroscopy is a valuable tool for characterizing carbon nanomaterials (CNMs) such as graphene, carbon nanotubes, and fullerenes. The technique allows researchers to non-destructively analyse CNMs, offering valuable information for quality control and optimizing their synthesis processes. With its sensitivity to subtle structural variations, Raman spectroscopy plays a crucial role in the comprehensive characterization of carbon nanomaterials in both research and industrial applications. Fig. 3 shows Raman Spectroscopy data for given CNM peaks at 1577 cm^{-1} and 1338 cm^{-1} indicating the graphitic and amorphous nature of obtained CNM.



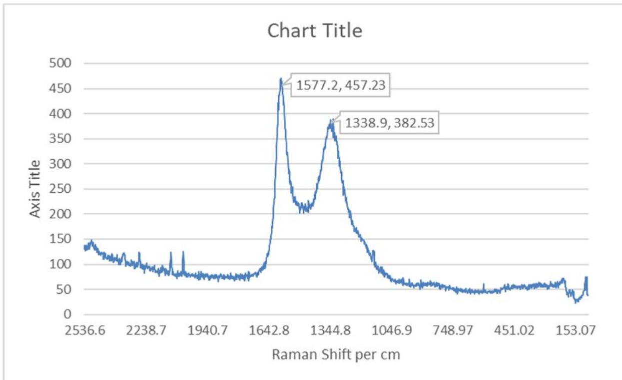


Fig 2. XRD plot for CNM treated with Nickel and Aluminium Fig 3. Raman Spectroscopy data

3) Energy Dispersive X-ray Spectroscopy (EDAX)

EDAX is a characterization technique that accompanies scanning electron microscopy (SEM). It analyses the X-rays emitted by a sample when bombarded with an electron beam. This provides information about the elemental composition of the material, Fig. 3 shows the spot of material that is used for EDAX, Fig. 4(a) shows graphical data and Fig. 4(b) shows the numerical values of elemental composition which confirms the presence of Ni and Al particles decoration.

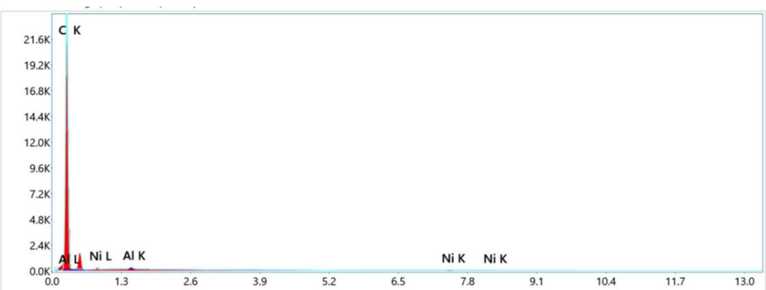


Fig 4(a). EDAX graphical data for metal decorated CNM

Element	Wt%	Atomic%
CK	99.6	99.9
Ni K	0.1	0.1
Al K	0.3	0.1

Fig 4(b). EDAX data for metal decorated CNM

4) Hydrogen adsorption study by bimetal decorated CNMs

Synthesised metal decorated CNMs were used for hydrogen storage study using Sievert's apparatus, Sievert's apparatus is a scientific instrument used to study the absorption and desorption of hydrogen in solid materials. Hydrogen adsorption value is 8.08 wt% at room temperature. This study reveals despite the relatively low obtained CNM exhibits a remarkable hydrogen adsorption capacity. This observation is attributed to the spillover effect. The spillover effect occurs when hydrogen atoms dissociate on the surface of one metal and then migrate to a neighbouring metal with a stronger binding affinity for hydrogen. In this case, the bimetallic decoration on the CNM surface provides active sites for hydrogen dissociation, while the underlying CNM structure possesses a strong binding affinity for hydrogen atoms. This synergistic combination leads to the observed high hydrogen adsorption even though the overall surface area of the material is not exceptionally large.

IV. CONCLUSION

This study concludes that, despite its relatively low surface area, CNMs decorated with bimetal combinations of Aluminium and Nickel nanoparticles exhibit a remarkably high hydrogen adsorption capacity 8.08 wt% at room temperature. This is attributed to the spillover effect, where hydrogen molecules preferentially bind to active sites on the CNM and then spillover onto the surrounding support material, significantly increasing the overall hydrogen uptake, by unravelling the underlying mechanisms and recognizing the importance of factors beyond just surface area, this study opens doors for the development of novel and efficient hydrogen storage solutions. Optimizing the bimetallic composition could lead to even higher hydrogen adsorption capacities, making these materials even more attractive for practical applications.

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