



## Macromolecular Nanotechnology

## Synergistic effect of vanadium pentoxide and graphene oxide in polyvinyl alcohol for energy storage application



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## ABSTRACT

A novel ultra-high-*k* composite material comprising of polyvinyl alcohol (PVA) as a polymer matrix and vanadium pentoxide (V<sub>2</sub>O<sub>5</sub>) and graphene oxide (GO) as fillers have been developed successfully using colloidal processing technique. The PVA/V<sub>2</sub>O<sub>5</sub> and PVA/V<sub>2</sub>O<sub>5</sub>/GO composites were characterized using Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, UV–vis spectroscopy (UV), X-ray diffraction (XRD), thermogravimetric analysis (TGA), polarized optical microscopy (POM), scanning electron microscopy (SEM) and atomic force microscopy (AFM). FTIR studies indicate the strong chemical interaction between GO and polymer matrix. SEM results confirm that GO was homogeneously dispersed within the polymer matrix. The dielectric measurements show that PVA/V<sub>2</sub>O<sub>5</sub>/GO composites exhibit ultra-high dielectric constant and low dielectric loss. The dielectric constant increases from ( $\epsilon = 189.40$ , 50 Hz, 40 °C) for PVA/V<sub>2</sub>O<sub>5</sub> (95/05 wt/wt) composites to ( $\epsilon = 5610.76$ , 50 Hz, 50 °C) for PVA/V<sub>2</sub>O<sub>5</sub>/GO composites with 2.5 wt% GO loading and the dielectric loss increases from ( $\tan \delta = 3.76$ , 50 Hz, 80 °C) for PVA/V<sub>2</sub>O<sub>5</sub> (95/05) composites to ( $\tan \delta = 9.77$ , 50 Hz, 150 °C) for PVA/V<sub>2</sub>O<sub>5</sub>/GO composites with 2.5 wt% GO loading. This study opens up the avenue to prepare novel graphene-based polymer composites having ultra-high dielectric constant and low dielectric loss and extends the application window of graphene based fillers.

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## 1. Introduction

Graphene is a new class of next generation material which consists of a monolayer of sp<sup>2</sup> hybridized carbon atoms arranged in a honeycomb crystal lattice. Graphene is known as a basic building block for other carbon materials such as zero-dimensional fullerene, one-dimensional carbon nanotubes (CNT's) and three-dimensional graphite [1]. Graphene has been studied extensively in many areas of science and engineering because of its unprecedented physical and chemical properties. The unique structure of graphene holds great promise in potential applications which includes nanoelectronics, sensors, batteries, supercapacitors, hydrogen storage and nanocomposites [1]. Graphene is reported to have high surface area

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(2630 m<sup>2</sup> g<sup>-1</sup>), Young's modulus (1 TPa), fracture strength (130 GPa) and the thermal and electrical conductivity of 5 W mK<sup>-1</sup> and 720 S m<sup>-1</sup> respectively [2,3]. Great efforts have been made in preparation of highly conducting composites using the solution-processed graphene for transparent electrodes [4–7] and photovoltaic device applications [8,9]. Bulk quantities of graphene can be produced by the chemical reduction of graphene oxide (GO) using graphite as a precursor material [10]. Since graphite is cheap and easily available, this chemical method for large-scale production of graphene is likely to be the simplest and most effective approach. GO has also attracted a great deal of attention due to its unique structure and properties. GO consist of a two-dimensional sheet of covalently bonded carbon atoms having various oxygen-containing functional groups such as hydroxyl, carbonyl and epoxide. The epoxy and carbonyl functional groups are attached above and below basal planes and carboxyl groups are located at the edges [5,11]. These functional groups make GO strongly hydrophilic in nature which helps GO to readily disperse in water as individual sheets to form a homogeneous dispersion. Also, GO acts as interfacial linkers which facilitate the stress transfer from polymer to GO [12]. It has been reported that the thermal stability [13–16], mechanical properties [17,18] and electrical properties [19,20] of GO reinforced polymers could be greatly improved due to its large aspect ratio, high strength, high Young modulus and strong interfacial interactions with polymers.

Vanadium pentoxide (V<sub>2</sub>O<sub>5</sub>) is a typical n-type semiconductor having lamellar layered structure. V<sub>2</sub>O<sub>5</sub> is one of the most stable oxides in the V–O systems having an energy bandgap of about 2.2 eV and it shows metal–semiconductor transitions which imply the sudden change in their optical and electrical properties. V<sub>2</sub>O<sub>5</sub> has many potential applications such as in field effect transistors, ultra-sensitive gas sensors, piezoelectric material, catalyst, hydrogen storage material, chemical sensors and biosensors [21,22] and has been studied as an electrode material for rechargeable lithium batteries because of its low cost and high energy density [23–25]. V<sub>2</sub>O<sub>5</sub> can be obtained in various nanostructures such as nanodisks [26], nanorods [27,28], nanowires [25,29] and nanotubes [30]. Recently, Liu et al., have reported V<sub>2</sub>O<sub>5</sub> nanowire/graphene composites for supercapacitor applications [31]. Similarly, Rui et al., studied porous V<sub>2</sub>O<sub>5</sub> sphere/graphene composites which show improved high rate performance [32]. Wang et al., synthesized V<sub>2</sub>O<sub>5</sub> nanosheet/graphene composites using the thermal decomposition of mixed precursors showing improved capacitive performance [33]. However, there are only a few experimental studies have been reported on polymer/V<sub>2</sub>O<sub>5</sub> and polymer/V<sub>2</sub>O<sub>5</sub>/graphene composites but the studies on the dielectric properties of polymer composites based on V<sub>2</sub>O<sub>5</sub> and GO have not been reported so far to the best of our knowledge. The layered structures of V<sub>2</sub>O<sub>5</sub> and GO could be beneficial in improving the dielectric behavior of the polymer matrix. Keeping this in mind, in the present investigation, the dielectric behavior of PVA/V<sub>2</sub>O<sub>5</sub>/GO composites was studied to highlight the novelty in the dielectric properties of proposed composites.

Polyvinyl alcohol (PVA) is the largest synthetic polymer produced in the world. It is synthesized by hydrolysis of polyvinyl acetate (PVAc) with various degrees of hydrolysis. The reactivity of hydroxyl groups of PVA depends strongly on the content of the residual acetyl group and the degree of hydrolysis [33,34]. PVA was chosen as a polymer matrix in this study because it exhibits high thermal and chemical stability, good flexibility, high tensile strength, non-toxicity and excellent film-forming ability [35–39]. PVA possesses high dielectric strength (>1000 kV/mm) and good charge storing capacity and it has been used extensively as a paper coating agent, in drug delivery as an adhesive carrier, in industrial formulations as a protective colloid and thickener and also in biomedical, textile and packaging applications [40]. It is quite appropriate to prepare PVA/GO composite films using a simple and eco-friendly method, considering that PVA has excellent water solubility and GO can be readily dispersed and fully exfoliated in water as individual graphene sheets to form a homogeneous dispersion [41,42]. The oxygen-containing functional groups of GO are feasible and effective to promote complete exfoliation and uniform dispersion of GO into the polymer matrix and to improve the interfacial bonding between them. The full utilization of GO sheets in polymer nanocomposite applications will inevitably depend on the ability to achieve full exfoliation and homogeneous dispersion of GO in polymer matrices [42].

In the present investigation, we prepared PVA/V<sub>2</sub>O<sub>5</sub>/GO composites using colloidal processing technique to take the advantage of both V<sub>2</sub>O<sub>5</sub> and GO and to develop ultra-high-*k* composite material. To the best of our knowledge, a study concerning the fabrication of ultra-high-*k* composite materials consisting of both GO and V<sub>2</sub>O<sub>5</sub> together has not been reported in the literature so far. With this interest, PVA/V<sub>2</sub>O<sub>5</sub>/GO composites were prepared and the dispersion state of GO and V<sub>2</sub>O<sub>5</sub> into polymer matrix was evaluated using FTIR, Raman, XRD, optical microscopy and SEM analysis. In addition, the dielectric properties of PVA/V<sub>2</sub>O<sub>5</sub>/GO composites were investigated using impedance analyzer at various frequencies and temperatures.

## 2. Experimental procedures

### 2.1. Materials

Polyvinyl alcohol (PVA) of molecular weight 85,000–124,000 g/mol and degree of hydrolysis 87–89% was purchased from Sigma–Aldrich, India. Natural graphite powder used in this study was supplied by Carbotech Engineers, Jaipur, India with a particle size of about 40 μm. The V<sub>2</sub>O<sub>5</sub> powder was supplied by Yogi Dye Chem Industries, Ghatkopar, Mumbai, India. Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), potassium permanganate (KMnO<sub>4</sub>), sodium nitrate (NaNO<sub>3</sub>) and 30% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) were purchased from S.D. Fine Chemicals, Mumbai, India. All the chemicals were of analytical grade and used without further purifications. Deionized water was used as a solvent for the preparation of PVA/V<sub>2</sub>O<sub>5</sub>/GO composites.

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