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# **A brief review on nanocomposites, their synthesis and potential applications in current scenario**

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**Abstract**---During past few years, studies in the field of nanomaterial sciences were more focused on nanocomposites due to their extraordinary and unexplored interesting properties. Our prime focus in this review is to address recent advancements in their manufacturing, processing, characterization of their functional attributes and also to explore the thrust areas of their utilization. In this way, we objectively explored and analyzed different areas such as aerospace, electric and manufacturing, defense and biomedical by addressing the strength and uses of nanocomposites respectively. Along with this, we also explored the area of biomedical sciences specifically their application in tissue engineering using zirconium oxide nanoceramic modified chitosan as an exemplary nanocomposite. Conclusively, this review will provide critical issues in nanocomposites research with promising functional utility to a greater extent.

**Keywords**---nanocomposites, chitosan, zirconium oxide, Ag<sub>2</sub>O/C-dots, PANI.

**Introduction**

Enhancing the complex nature of nanomaterials in contexts of composition and structure has piqued the interest of researchers because it has the potential to produce novel scientific outcomes as well as significant improvements in practical

utility [1]. Several approaches to the synthesis of nanostructured composites are being developed. Commonly, composites are made up of two materials which don't form specific stoichiometric compounds, whereas, manual variation in the stoichiometry of two component can further increase their specific properties. A high degree of integration capability of even single composite component can replace the several metallic components in final structure [1]. So, it's very easy to manufacture very neat and shaped composite materials in a short duration. This essentially brings a strong prominence in the manufacturing of nanocomposites in last 2 decades.

Here, we would like to focus on the nanomaterials integrated into polymer matrix for producing the complex nanocomposites. The complex of nanomaterials with polymer matrix leads to significant changes in the properties such as mechanical strength, electrical and optical properties [2]. As a result, final product has the superior properties as compared to individual constituents. This change in properties is due to very less amount of material normally between 0.5 to 5 % by weight. The products so formed can be of macro or micro size but there should be one nano size dimension filler material [2].

There are various methods and techniques which have been discovered for the characterization of nanocomposites essentially to predict various supreme properties by modeling and simulation of their structures [3]. These techniques further help in exploration of surface structures to an extent atomic resolution. Using one of these methods, Toyota Central Research Laboratories, Japan claimed improvement in mechanical and thermal properties through nano fillers using a NYLON-6 nanocomposite [3]. While, many nanocomposites exist in nature such as abalone shells, bones, wood etc. These can be the mixture of calcium carbonate or calcium phosphate along with a biopolymer which naturally exist which justify that Nature is the master chemist with incredible talent [3]. The properties of nanocomposites also depend on morphology and interfacial characteristics of raw materials [3].

Why nanoscale variation essentially required as of microscale:

- ❖ Polymer nanocomposites have exceptionally high surface area/aspect to volume ratio as prudent in earlier findings and mentioned in figure 2. The fillers of 1D, 2D or 3D used in formulations of polymer nanocomposites such as particles, tubes, fibers, wires, thin film coatings, quantum wells and fullerenes [5-7].
- ❖ The area of interface between the reinforcing material and matrix is generally higher than the conventional composite material.
- ❖ Reinforcing agents are commonly carbon black, alumina, silica nanoparticles and polyhedral oligomeric silsesquioxanes (POSS) [8].
- ❖ There is an improvement in mechanical properties such as increase in ductility with no decrease in strength while increase in scratching resistance.
- ❖ It also leads to enhancement in optical properties.
- ❖ The micrometer size particles act as stressor while the small structure of nanocomposites doesn't elicit stress.

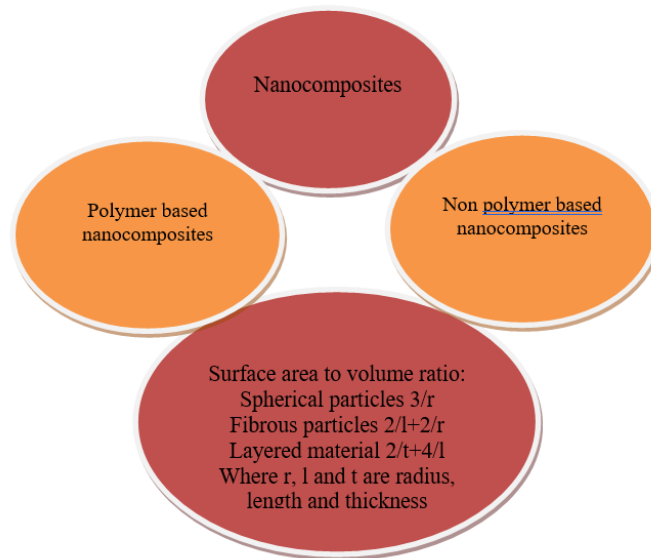


Figure 2: Aspect ratio of polymer nanocomposites

Due to the multifunctionality that nanotechnology utility even different governmental setups raised funding to establishment of *National Nanotechnology Initiative* [9]. Funding based opportunities have modeled manufacturing processes and improved nanoproducts in more significant ways. That further increase the scope for the nanocomposites in various research areas such as communication, data storage, solid oxide fuel cells, defense applications, health and medicine, energystorage, catalysis, gas separation, filtering biochemical separations, battery and capacitor elements. They may pose significant utility in the field of environmental, transportation, aerospace and marine sciences [9-12].

### Synthesis of Nanocomposites

There are the various techniques for the synthesis of conventional composites such as wet lay-up, pultrusion, vacuum assisted resin transfer molding (VARTM), resin transfer molding (RTM), filament winding, autoclave processing, and prepreg method etc. [13]. The main difficulty in processing of nanocomposites is the large amount of surface charges because of those nanocomposites get agglomerate and the moments also gets agglomerated. This essentially affects the functional properties due to unequal distribution of charges in each direction. Secondly, the density of nanoparticles is very less so the dispersion of the particles in the bulk also becomes difficult [13-15]. So, due care should be taken while synthesizing nanocomposites. Different methods are being used for the synthesis of nanocomposites as described below:

**Solution casting:** In this method the polymer, solution and nano reinforcement are mixed thoroughly by using ultrasonication, followed by nanocomposites thin film formation after evaporation of the solvent or solution. These solution and solvent used will help in intercalation and mobility of polymer chains. The adequacy of solution casting depends on its application. This method enables the

addition of more concentrated thermally conductive fillers more than feasible with melt extrusion process. By this method, we can synthesize a film of thickness ranging from 12 to 150 micron [16].

*Melt blending:* Polymer and nano reinforcement are extruded and then pressed using a dye to get a nanocomposite. Polymer mobility comes from thermal energy. This method formed the exfoliated nanocomposites structure and showed higher gas barrier and creep resistance than solution casting method and melts blending films respectively. The physical performances of nanocomposite films formed by this method are superior than solution casting method [18-19].

*In-situ polymerization:* This is basically of two types, one process is intercalation and another is exfoliation. Here, we are describing one after another based on their relative relevance and advancements. Firstly, intercalation technique polymer can be formed in between the intercalated sheets that are essentially the reversible inclusion of molecules into the layered structure [20]. Each layer of the structure formed is of very minute nanometer size. In this regard, manufacturing of nylon-montmorillonite nanocomposite was the first successful product which was approached by this technique followed by many other thermoplastics [21]. For this purpose the layered silicate mineral is swollen in monomer and then the polymerization of the monomer initiated by radiation or heat or an initiator [22-25]. Different studies reported use of intercalation method for the synthesis of nylon-6 EG nanocomposites [26], expanded graphite [27], and graphite nanosheets by the sonication of expanded graphite and dispersed into polystyrene matrix [28].

Exfoliation is the extreme case of intercalation. It includes the complete separation of layers which leads to the emulsion polymerization. This method was first used by Toyotaresearchgrouptoproducepolyimidenanocomposites. The phase homogeneity of exfoliated nanocomposites is higher than the intercalated [29]. Several factors are need to be considered while the formation of exfoliated nano clay reinforced composites like temperature, time, viscosity, functionality of resin matrix, curing agent, structure of clay and curing time etc. The rate of polymerization is different in or out of the filler material so it is important to control intra or extra gallery polymerization for uniform polymerization reaction. Pinnavaia *et al.* [30] developed the exfoliated epoxy resin. Thostenson *et al.* [31] stated the properties between exfoliated graphite platelets and clay platelets belonging to different literatures.

*Electro-spinning:* This method can be used in preparation of composite nano fibers from nanometer to micrometer range. First of all, a mixture of polymer matrix and graphene is made in a solvent commonly N, N-dimethyl formamide (DMF) [32]. After that electrospinning of mixture is done by applying high voltage on a syringe needle. The interested properties are being presented by the electro spun fibers such as low density, high pore volume, high surface/volume ratio and outstanding mechanical strength.

*Electrodeposition:* This technique uses the electrochemical reaction for the preparation of nanocomposites. It consists of three electrodes: working electrode, reference and counter electrode, which is usually made of platinum [33].

Electrically conductive material is placed on the working electrode where the nanocomposite film is formed. Electrodeposition takes place at specific potential and stops after significant amount of charge have passed [34].

### **Applications of nanocomposites in different areas**

The numbers of applications of nanocomposites have been growing at a rapid rate. A range of polymeric nanocomposites are used for biomedical applications such as tissue engineering, drug delivery, cellular therapies. Figure 1 described a brief of concerned areas for the potential use of nanocomposites.

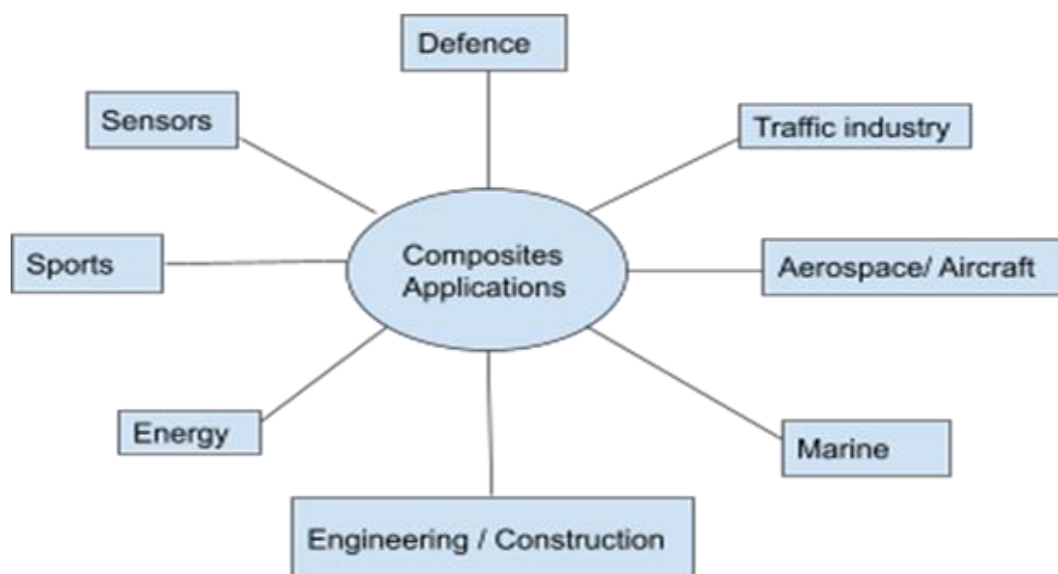


Figure 1: Different areas of composites applications

### **Applications of nanocomposites in different areas**

Formation of zirconium oxide doped chitosan-hydroxyapatite nanocomposites for bone tissue engineering: - Mechanical strength and porosities of the organic and inorganic hybrid nanocomposites i.e., chitosan-polyethylene glycol-nanohydroxyapatite based composite become similar to the human spongy bone because of incorporation of zirconium oxide nanoparticles ( $ZrO_2$  NPs), even 0.1 - 0.3 wt.% of ( $ZrO_2$  NPs). Antimicrobial effects were also observed against the gram-negative and gram-positive bacterial strains [35].

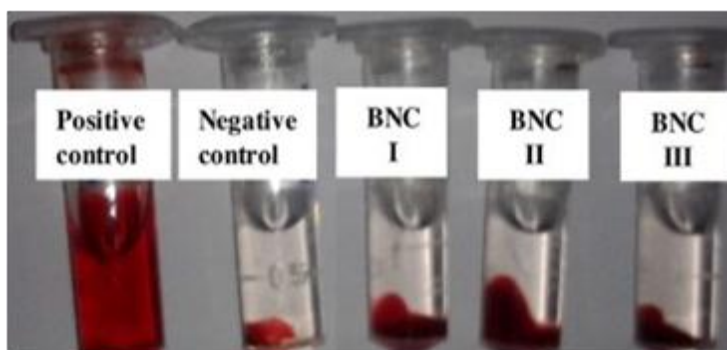


Fig. 3: Hemolytic assay of BNC I-III

As shown in Fig. 3, the blood compatibility of prepared nanocomposites with addition of 15 wt.% of HA- ZrO<sub>2</sub> in CS-PEG (55:30 wt.%) was investigated, which shows that the cytocompatibility, porosity, and antimicrobial activity further increased. The water absorption capacity and compatibility with human blood & pH were also enhanced. The mechanical properties required for new bone formation, and porosity allows cell migration & nutrient exchange. Cytocompatibility helps in cell multiplication and to eliminate the bacterial infection, hence improved the antimicrobial properties. Incorporation of HA into CS enhanced the brittleness of the CS-HA composite due to poor mechanical strength [36].

Therefore, this composite had been incorporated with polyamide, polyethylene glycol or polycaprolactone to increase the solubility and flexibility. Among these mentioned compounds, the PEG has excellent water solubility, flexibility, biocompatibility, and ability to exude from the body without forming toxic metabolites [37-38], which makes it better candidate. CS-HA-PEG composite fabricated by Shakir *et.al* found compressive strength of 6.7 MPa while that of CS-HA composite has 5 MPa and with the incorporation of zirconium oxide in CS-HA-PEG nanocomposite the mechanical strength has been increased to 15.83 MPa [39].

Synthesis of bone-like (CS-PEG-HA-ZrO<sub>2</sub>) nanocomposite (BNC I- III): ZrO<sub>2</sub> nanoparticles were manufactured by adding an aqueous solution of NaOH in zirconium oxychloride octahydrate solution, which was prepared in methanol and water having a 1:1 ratio at 5°C [40-43]. The wet chemical precipitation method was used to synthesize nano HA. Different samples of CS-PEG-HA-ZrO<sub>2</sub> nanocomposites were prepared by varying weight percentages of CS-PEG in nano HA-ZrO<sub>2</sub>. Firstly, the solution of CS was made in 85% formic acid solution then PEG was added. This solution is mixed with HA-ZrO<sub>2</sub> and a film was prepared by adding into the Petri-dishes.

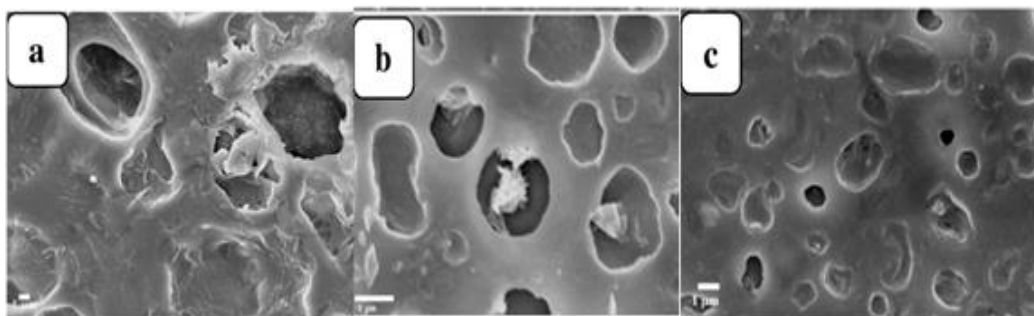


Fig. 4: SEM images of [a] BNC I [b] BNC II [c] BNC III

Characterization of these composite were done by FTIR & X-ray diffraction. The size and morphological analysis were done by FESEM, TEM combined with SAED was used to determine crystalline and surface morphology as depicted by fig. 4. The porous structure has been divulged by SEM images having a pore size of less than 1–10 $\mu$ m. The porosity of BNC III was found to be the greatest of three. It had been increased from BNC I (65.71%) to BNC III (73.02%). The increased water absorption power could be due to ZrO<sub>2</sub> NPs hygroscopicity and pH was found to be the same that of blood plasma i.e., 7.4 [44].

Detection of Fe (III) ions by silver oxide/C- dots nanocomposite: Metal ions are fundamental elements which play a crucial role in biology, catalysis, medicine and environment [45]. Therefore, the sensing of metal ions has become significant. The design and development of fluorescent sensors are very much prominent. Iron plays a very important role as an electron and oxygen carrier, the deficiency of iron causes many diseases like anemia, Alzheimer's and Parkinson's disease, tiredness and decrement in work activity and immune system get affected [45-46]. Also, the excess of Fe<sup>3+</sup> ions damages nucleic acids, lipids & proteins [47]. So, it becomes important to develop a reliable method for detection and quantification of Fe<sup>3+</sup> ions. The Ag<sub>2</sub>O/C- dots get excited at the wavelength of 370 nm and emit at a wavelength of 455 nm. It can give rise to a reliable method to quantify Fe<sup>3+</sup> ions because of quenching of this eminent emission by Fe<sup>3+</sup> ions [48]. Over the last few years, heavy metal-based semiconductor quantum dots have been used as fluorescent probes as these have many advantageous properties such as wide excitation spectrum, narrow emission spectrum, brightness and photochemical stability [49-51]. But, they are highly toxic to the environment. Now with the development of carbon dots (C- dots) as a replacement to conventional semiconductor dots has supplemented the research interest in material science. Due to the emergence of C- dots, there has been a great advancement in various fields such as photonics, biosensing, electronics, catalysis, medicine and imaging [52-55].

Their less-toxicity, magnificent biocompatibility, fluorescence, photostability, non-blinking behavior, high aqueous solubility, easy surface functionalization, resistance to environmental change, robust chemical inertness and ease of preparation has amplified the interest in optoelectrical appliances and assemblage as composite material [56]. This makes the growth of metal or metal oxides viable on the surface of C-dots. Metal/Metal oxide enhances the fluorescence intensity of C - dot, this is known as metal enhance fluorescence [57]. Zhang *et al.* reported

the C-dots doped with Ag@SiO<sub>2</sub> core shell nanoparticles shows the metal enhanced resonance which attributed to the surface plasmon resonance [58]. A fluorescent “turn- on” method was reported by Amjadi *et al.* for analysis of cysteine using C- dots/Ag nanoparticles [59]. Shen *et al.* divulged the growth of silver nanoparticles on the surface of C- dots by a single-step approach [60]. C - dots reported are synthesized by hydrothermal assisted photoreaction & have a size less than 10 nm with abundant oxygenous and hydrogenous residues [61]. The average size of nanocomposite obtained was 2.8 nm by dynamic light scattering (DLS).

The detection of Fe<sup>3+</sup> ions done by the Ag<sub>2</sub>O/C- dots is based on the spectrofluorometry. The quenching of the intensity of light follows the stern volmer equation:

$$F_0/F = 1 + K_{sv}[Q] \quad (1)$$

Where  $F_0$  &  $F$  is intensity of fluorescence in absence and presence of quencher respectively,  $Q$  is concentration of Fe<sup>3+</sup> ions i.e., quencher.  $K_{sv}$  is stern volmer quenching constant. Presence of other metal ions do not affect the selectivity and specificity of Ag<sub>2</sub>O/C-dots towards Fe<sup>3+</sup> ions [62] as given by plots of fig. 5.

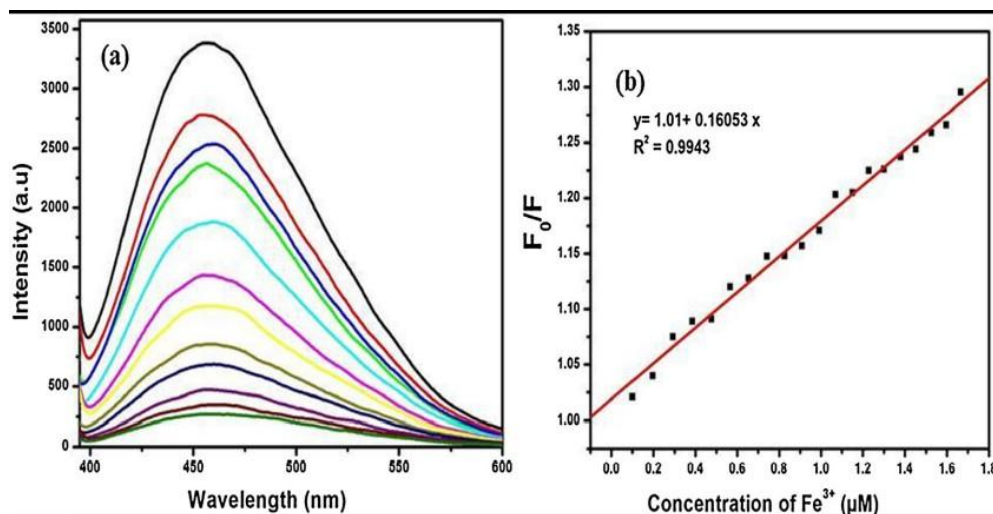
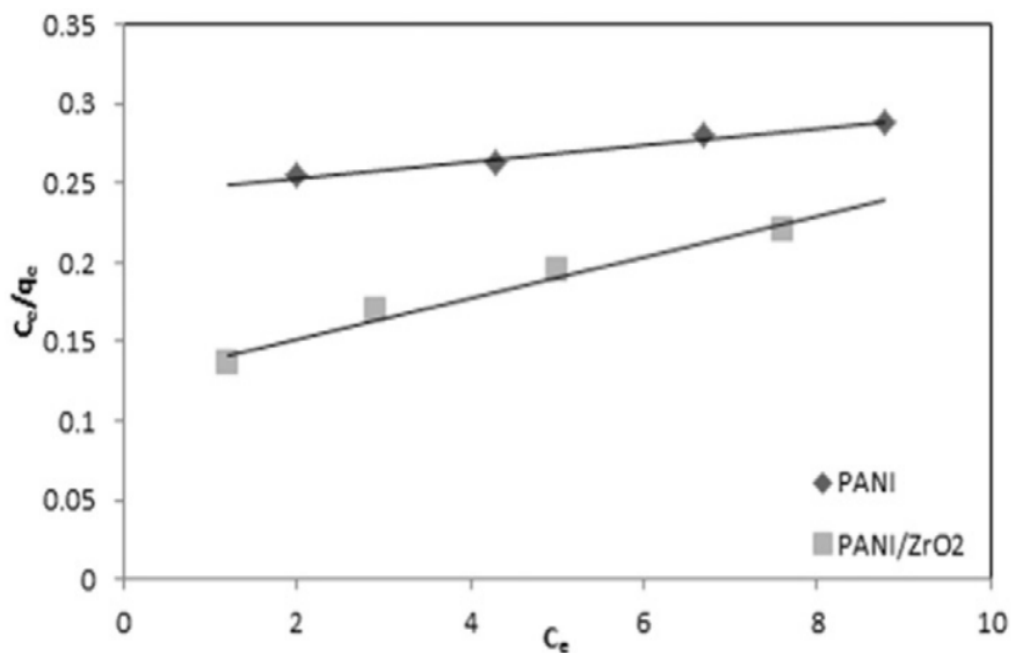


Figure 5: (A) Fluorescence emission spectra of Ag<sub>2</sub>O/C- dots with varied concentrations of Fe<sup>3+</sup> ions. (B) Stern - Volmer plot for fluorescence quenching of Ag<sub>2</sub>O/C- dots by Fe (III) ions.

Polyaniline modified with ZrO<sub>2</sub> and polyaniline nanocomposite for dye adsorption application: It has been evaluated that approximately 40,000 tons of dyes, out of roughly 450,000 tons of productivity released out in waste water which is not being used and toxic for aquatic life [63]. Some of these dyes have virulent and even cancer-causing effect. These are responsible for many health problems like, methylene blue leads to several cardiovascular diseases as hypertension and

precordial pain. It is also responsible for dermatological infections, gastrointestinal diseases and abdominal pain [64]. So, the discharge of these poisonous dyes into water not only hazardous to the environmental but also not good for human and aquatic life[65]. It cannot be vanished by biodegradation alone. The most important physical process for their removal is adsorption. There are various materials used as adsorbent but PANI / PANI ZrO<sub>2</sub> is found to be quite effective for the adsorption of methylene blue. PANI has good environmental stability, very cheap to synthesize and also has the anticorrosive property. Langmuir and Freundlich isotherms were plotted to describe the properties of adsorbent. And the effect of temperature on the adsorption was also noted & it was found to be endothermic process.



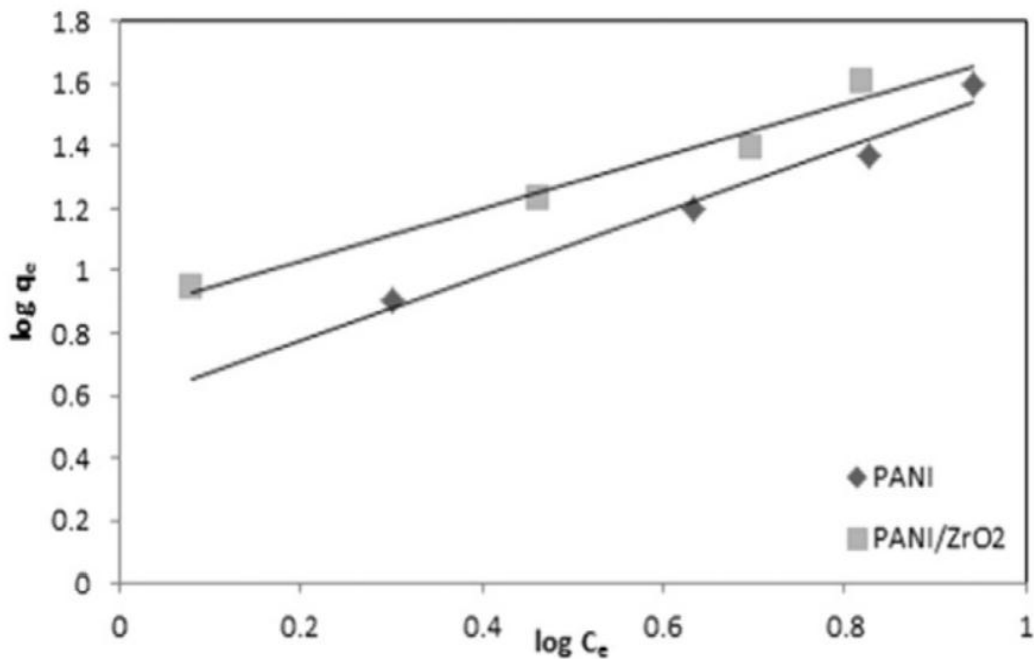


Figure 6: [a] Langmuir plot for the adsorption of MB by PANI and PANI/ZrO<sub>2</sub>  
[b] Freundlich plot for the adsorption of MB by PANI and PANI/ZrO<sub>2</sub>.

The Langmuir equation can be written as,

$$\frac{C_e}{q_m} = \frac{1}{K_L q_{\max}} + \frac{C_e}{q_{\max}} \quad (2)$$

$C_e$  is the concentration of MB at equilibrium  $q_m$  is the adsorbed concentration,  $K_L$  is the Langmuir constant and  $q_{\max}$  is the adsorption capacity [66].

Also, the Freundlich equation can be written as [67]

$$\frac{x}{m} = Kp^{1/n} \quad (3)$$

It is also written as

$$\log \frac{x}{m} = \log K + \frac{1}{n} \log p \quad (4)$$

$$\text{Or } \frac{x}{m} = Kc^{1/n} \quad (5)$$

It is also written as

$$\log \frac{x}{m} = \log K + \frac{1}{n} \log c$$

(6)

Where 'x' mass of adsorbate 'm' mass of adsorbent,  $p$  is equilibrium pressure of the adsorbate,  $c$  is equilibrium concentration of adsorbate,  $K$  and  $n$  are constants for a given adsorbate and adsorbent at a given temperature.

Nanocomposites applications in lithium-ion batteries: In today's world we need the high- performance batteries for the communication devices, electric/ hybrid vehicles and portable electronic devices [68]. Due to the high energy density, high voltage and light weight, Li- ionbatteries are one of the most relevant and satisfactory module. But there are somewhat safety issues also, as there is growth of lithium dendrites during the charge/ discharge cycling. For the high performance of batteries, electrodes are the main determining factor. Scientists have made the numerous efforts to explore different anode materials in order to increase the energy density of these batteries. Traditional graphite materials have been replaced by various metals, metal oxides and metal sulfides since the commercial graphite anode has the theoretical capacity of 372 mA hg<sup>-1</sup> only [69].

Metal oxides have the theoretical capacities compared to that of carbonaceous material. There is huge volume variation and considerable mechanical strength while charging/ discharging process of these materials, which lead to the electrode disintegration and rapid capacity fading [70]. Out of the metal oxides, TiO<sub>2</sub> was considered up to the mark for anode material as it can be operated at high voltage, which preserve the growth of lithium dendrites. Also, it is ecofriendly, cheap and shows low volume expansion during charge/ discharge cycles. But there are some drawbacks also like poor electronic conductivity, low Li-ion diffusion ability, and intrinsically low theoretical capacity [71]. But this can be overcome by making composite nanostructured TiO<sub>2</sub>-SnO<sub>2</sub>-C ternary systems. A new nanostructure of SnO<sub>2</sub> nanoparticles (NPs) confined in hollow TiO<sub>2</sub> nanowires with a reversible capacity of 445 mA h g<sup>-1</sup> at 800 3 mA hg<sup>-1</sup> was synthesized by *Chen group* [72].

Ge nanoparticles/graphene (Ge NPs/GR) nanocomposites exhibits the capacity retention of 90% after 15 cycles. Due to their huge theoretical capacities, Si 4200 mA h g<sup>-1</sup> and Ge 1600 mA h g<sup>-1</sup> had attracted the interest in field of lithium-ion batteries. But, Ge was found to exhibit higher diffusivity of Li and lower volume variation as compared with Si. Maier *et al.* synthesized carbon matrix supported Ge NPs [73].

SnO<sub>2</sub> becomes more captivating due to high theoretical reversible Li<sup>+</sup> storage capacity which was calculated to be 782 mA h g<sup>-1</sup>. Integrating SnO<sub>2</sub> with carbon material reduces the strain due to volume expansion during charge/ discharge cycle. Graphene is suitable for this purpose due to the excellent mechanical and electrical properties to make high performance composite with anode material. Graphene is generally prepared by reducing exfoliated graphene oxide sheets. SnO<sub>2</sub> incorporation not only prevent the aggregation of reduced graphene oxide

sheets but also enhance the electrochemical performance of synthesized anode material [74]. Similarly,  $\text{Co}_3\text{O}_4/\text{RGOs}$  shows the enhanced capacity as compared to that of  $\text{Co}_3\text{O}_4$  as anode material in Li ion batteries. This good electrochemical behavior is due to increase in surface area for the absorption of more Li ions. This way relevant materials are synthesized by scientists for the high performance of batteries [75].

## Conclusions

In summary, we have discussed about the synthesis of nanocomposites by several processes. And the result of the addition of nanoparticles is a drastic improvement in properties of composite materials. As we have seen some of the applications of nanocomposites as biomedical probes, as sensing probes, as an adsorbent and in batteries to increase its working capacity. We have seen that zirconium oxide doped chitosan-hydroxyapatite nanocomposites can be devised for bone tissue engineering and silver oxides/C-dots as biomedical sensing device with high selectivity for Fe (III) ions. Polyaniline modified with  $\text{ZrO}_2$ -polyaniline are developed for the hazardous dye adsorption. Attractive stable cycle performances can be observed by the incorporation of the crystalline nanoparticles in composite materials. In this way nanocomposites can be used in multipurpose way.

## Author Contributions

Concept, design and overall supervision: SW; Data Acquisition: SW, drafting of the manuscript: SW wrote and edited the manuscript with inputs from SA and NK; all authors provided comments on the manuscript.

## Conflict of Interest Disclosures

Authors have no conflict of interest to declare.

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