

Study of high-performance polymer matrix nanocomposite: A Review

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Abstract - Nanomaterials have piqued researchers' interest in the last ten years because of their distinctive physical and chemical features, which have led to the creation of new construction materials with innovative uses. Compared to micron fillers, the use of silica nanoparticles greatly improved the breakdown strength and voltage endurance. The volume fraction and filler size of this composite are what give it its main advantages. One of the foremost alluded to and utilized cementitious nanosized materials is nano silica ($nSiO_2$). The downsizing of a certain electronic gadget uses nanomaterials. The need for a more practical, less expensive approach to generating superior materials, particularly silicon dioxide, arises from the new technological era. This paper covers a literature review on the various techniques of production of a nanocomposite's substance called $nSiO_2$ and its technological applications, along with the conditional suitability of the process.

Key Words: Nanocomposites, Silica, Sol-gel, Polymerization, Ball milling.

1. INTRODUCTION

The findings of study demonstrate that adding nano silica particles to a modified polypropylene polymer binder increases its resistance to fatigue. In a number of disciplines, such as chemistry, physics, and materials research, metal oxides are crucial. NEMS, MEMS, sensors, piezoelectric devices, coatings for corrosion passivation, gas cells, and catalysts in technical applications are all made with oxides [1-5]. Oxide nanoparticles can display distinctive physiological and chemical characteristics as a result of their confined measurement and an excessive density of corner or area floor locations. Because of this, transition metallic oxides (TMO) are most frequently employed in the rapidly evolving disciplines of electronics, magnetics, solar cells, photocatalysis, and gas sensors [2]. SiO_2 has established itself as a challenging study topic because of its exceptional physical and chemical features. Amorphous silicon dioxide is the most well-known crystalline form, followed by quartz, cristobalite, tridymite, stishovite, and coesite.

Silica powder has been used in the mechanical industry as well as precise structures for chemical catalysts, ceramics, and photo-electricity elements, among other things, because of its purity, shape, measurement, and

distribution. Due to its physicochemical advantages, the silica corpuscular powder's application fields are constantly expanding. The electrical spectra of the inter-band transition show more pronounced peaks in crystalline SiO_2 than in amorphous SiO_2 , and the power of the absorption component is about 1 eV higher [3, 4]. The sensitized mesoporous silica with acriflavine dye could be advantageous for nanosensors and nanolasers. [5]. SiO_2 can be used to increase the photocatalytic reproduction of oxide materials and polymers because of its high band gap, which increases the catalyst's accessible surface area [6, 7]. Micro-injection molding, in-situ polymerization, co-precipitation, sol-gel, and many other synthesis techniques are reportedly used for Nanocomposite materials [8, 9]. The leaching process, a chemical-based method, is used to create crystalline Nano silica powder. This crystalline nano silica powder is produced and then utilized as a filler in the creation of nanocomposite. According to the article, different fillers like SiO_2 , TiO_2 , MoO_3 , etc., allow the electromagnetic behavior of ferrite magnetic materials to be significantly modified. [10] These fillers encourage the catalytic process for adjusting the electromagnetic behavior.

Silica is a micronutrient that is crucial for plant growth and production in agriculture and is regarded as an essential micronutrient. Additionally, it strengthens the leaf, shoot, and root as well as increases weather tolerance, and lessens the detrimental impacts of some poisonous components in the soil. Silica can sterilize and kill fungi during the germination process, which boosts germination rates. And finally, nano silica (0–100 nm) can easily pass through the cell membrane due to its nanoscale and high surface area. As a result, utilizing nanomaterials, such as nano-silica, is thought to be a new area of research for soaking seeds. Natural or synthetic materials can be used to extract nano-silica. Several approaches, including the Stobr technique [11], sol-gel methods [12], and a water-in-oil nanoemulsion system, were used to create nano silica [13].

However, only a small number of investigations have been done using the delayed gelation approach and freeze-drying techniques [14].

The proper method of solution, a component of sol-gel, has the benefit of being simple to use, requiring little processing, and allowing for the modification of the

material's composition or the control of the purity and uniformity of the SiO₂ properties. Easy solution strategy reports are mentioned in references [15-17]. However, it is necessary to generate vast amounts of SiO₂ particles using a straightforward process and low-cost raw ingredients. In the present study, the SiO₂ particles were produced using cheap raw materials and the easy solution method.

2. Utilizing Polymer

Composites are man-made multiphase materials. It means that the two phases were individually existing, and then they were put together to form a composite material. Materials like phenolic, polystyrene, polyester, and vinyl were manufactured, but still, to obtain strength and rigidity, there is a need for reinforcement. Further, in 1930's development of resins was a very important milestone achieved in the industry of composites, and thereafter, within five to eight years, the world observed the development of glass fiber and fiber reinforced polymer (FRP) [18, 19].

As we know, composites are combinations of two-phase/constituents; these two constituents are generally mixed at the macroscopic level with the condition that they are not soluble in each other. These two constituents are namely reinforcing phase and matrix. Matrix is the one in which reinforcing phases like flakes, fibers, or particles are embedded, and it is generally continuous. The reinforcing phase is also called the discontinuous phase. However, both phases may be interrupted in some composites, such as sandwich composites. Composites can be divided into polymer matrix composites (PMC), metal matrix composites (MMC), and ceramic matrix composites (CMC) based on the matrix. Examples of polymer matrix composites from this category include GFRP (glass fiber reinforced plastic), CFRP (carbon fiber reinforced plastic), and others. The composites are also categorized based on the distribution and shape of the reinforcing phase as particles and fibers, whereas further fibers are classified as discontinuous (short fiber) and continuous (aligned or long). Advancement and utilization of modern composite (invention of resins with glass fibers) were started. Automobile bodies, boats, and aircraft are being started to form from this fiberglass. In 1938, other higher-performance resin systems like epoxies also became available. Thereafter, during World War 2, the researchers understood that fiber composites are transparent to radio frequencies, so this phenomenon can be helpful in planes/jets and avoid visibility, but before that, this was firstly implemented in commercial grade boat hulls. Now till 1960's researchers understood that because of the good stiffness-to-weight ratios of carbon fiber, they found their application in a variety of fields like sports, boats, aerospace, automotive, and even in consumer goods.

Two or more particles are added together, fibers or particles embedded into the matrix, and the preparation of the new material is nothing but composite material, which is the modern-day example of structural composite material. In the composite material, the matrix performs two supreme tasks, the first one is to bind the embedded fibers or particles, and the second is the distribution of force or stresses over embedded fibers. So based on the matrix, composite materials are get classified as shown in Figure 1 [20].

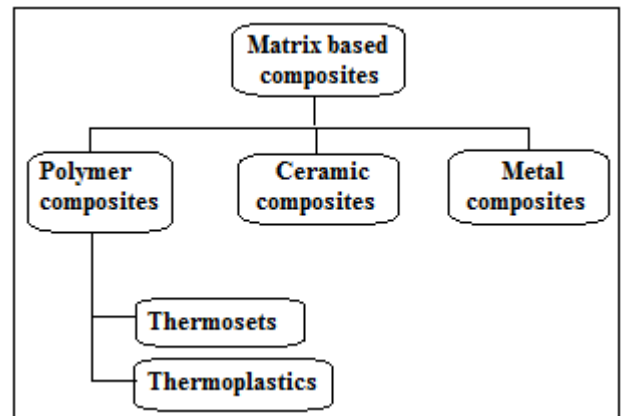


Fig. 1 Matrix-based classification of composites

The use of composite materials in various structures has grown in popularity over the past few decades, particularly in aerospace structures and ship hulls. Airframes, etc. Composite materials are chosen over traditional because of their advantages, particularly stiffness and strength. This is how epoxy works. Resins are frequently used in various matrix components, including engineering, because of their superior mechanical as well as insulating qualities.

The uniform dispersal and scattering of filler particles in the polymer matrix are essential for the enhancement of the mechanical characteristics of polymer matrix nanocomposites (PMNCs). In other words, a major issue in the production of PMNCs is inadequate nanoparticle dispersion and distribution in the polymer matrix. In a matrix, agglomeration normally declines as particle dispersion increases, i.e., more particle dispersion results in less agglomeration of particles. On the other side, the distribution describes how evenly the primary particles or their agglomerates are spread throughout the volume. [21-23]

PEK is a Poly-(ether ketone) polymer that appears in the form of a semi-crystalline polymer. It is thermoplastic and has good thermo-electro-mechanical and chemical properties, renowned for its suitable impact energy, fatigue electricity, and creep resistance. The glass transition temperature (T_g) of PEK is around 155-160°C. Because of this high glass transition temperature, it possesses good

dimensional stability and can be used for wide temperature ranges like the space shuttle, space antennas, flexible electronics, robotic systems, and many more [24-26].

One of the many techniques to increase the continuity between the hydrophobic polymer and hydrophilic nano silica is surface chemical treatment [27-29]. Utilizing a reactive modifier, such as silane coupling agents, can result in a strong interface and good dispersion. According to this theory, the coupling agent's organofunctional groups might be replaced by hydroxyl groups on the surface of nanoparticles while also reacting with polymer chains [30, 31]. Due to its superior thermo-mechanical stability, better resistance to chemical corrosion, and great tribological behavior, thermoplastic poly ether ether ketone has been used more and more in industrial applications.

The qualities of poly ether ether ketone (PEEK), including its great mechanical and thermal strength, stability, and resistance against corrosive conditions because of chemicals, make it one of the most significant engineering polymers. PEEK has been used as a biomaterial for orthopedic, trauma, and spinal implants more frequently since 1987 [32, 33]. According to in vitro biocompatibility tests, PEEK does not cause any mutagenic or cytotoxic activities [34]. PEEK has even been referred to as a bioinert material because it doesn't harm human tissues in any way and doesn't release any ions or other compounds either [35].

3. Process of preparation of nanocomposites:

This section describes the most recent developments in the mechanical milling of polymer matrix nanocomposites. An exploration of potential future circumstances that could be produced by the use of enhanced experimental techniques. Preparation of composites with PEEK was a challenging job. PEEK has a high melting point as well as has low dissolving capacity in solvents. So the possibility of heat treatment and homogeneous liquid mixing process get deducted from the process. Now only option that remained was solid-state polymer processing (using methods like mechanical alloying and solid-state shear pulverization, which do away with the heat and solvent issues of conventional approaches and offer processing flexibility and simplicity) is one way to handle this issue. A widely accepted MA instrument and tried-and-tested method for dealing with materials with tiny microstructures in both metals and ceramics is the planetary high-energy ball milling process [36-39]. Initially ball mill process was employed in 1994 by Namboodri et al. [40] to generate a polyamide/BaTiO₃ composite. In order to embrittle polymers and get rid of their viscoelastic properties, ball milling of polymers was modified below cryogenic temperature, which was produced by a cryogenic medium like liquid nitrogen. This technique is called as cryomilling

[36-38]. Cryomilling was employed in 1997 by Giri et al. [41] to create a polyethylene/Fe nanocomposite.

3.1 Planetary Ball Milling

Because very small quantities of precursor powders are utilized, planetary ball mills are typically employed in mechanical alloying. As a result, this method is frequently used in research labs. A planetary mill includes a turntable and four bowls. The turn disc rotates in the opposite direction from the bowls. The turn disc and bowl both rotate on their axes, creating centrifugal forces. The powder mixture is fractured and cold welded as a result of the centrifugal forces acting on the balls and the powder mixture. The impact energy of the milling balls in normal orientations can be up to 40 times larger than the acceleration caused by gravity. A planetary ball mill is a good alternative for high-speed/energy milling because of this [42].

Ball processing could be a mechanical handle frequently utilized to break up solid materials into minor pieces [43-45]. Within the routine approach, the reactants are ordinarily broken and separated utilizing dissolvable atoms; in any case, in ball milling, the reactants are broken using mechanical powers. Mechanochemistry may be a term that has as it was recently utilized [46]. In various survey articles [47-49], the utilization of ball milling within the blend and responses of natural compounds have been examined. Figure 2 shows the process of synthesis of nanomaterial. Solvent-free ball processing is once in a while utilized within the amalgamation of natural compounds.

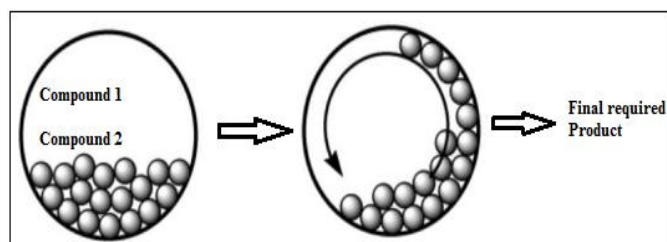


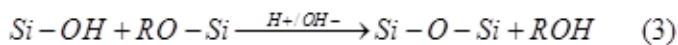
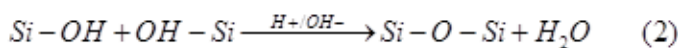
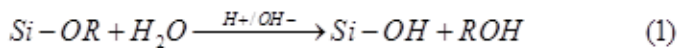
Fig. 2 Ball milling nanomaterial synthesis

3.2 Sol-Gel Process

Sol-gel reactions, a well-known method for creating inorganic glasses and ceramic precursors, can be used to produce these materials at relatively low temperatures. The fundamental advantage of the process is that ceramics are treated gently under low pressure and temperature conditions. In recent years, the sol-gel technique has been widely used to create innovative organic-inorganic composite (hybrid) materials, referred to, respectively, as "ceramers" and "ormosils" or "ormocers" [50, 51]. The sol-gel reaction is used to improve the adherence of organic molecules, which are frequently polymeric and include

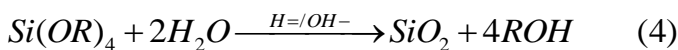
functional groups, to the ceramic-like phase in composites. This innovative reinforcement technique is very beneficial and may result in reinforcing particles within a polymer matrix. Additionally, because they are typically nanocomposites, these novel hybrid sol-gel materials have the potential to offer unique combinations of features that are not possible with other materials [52]. Numerous books [53–59] have provided thorough reviews of the research on the use of the sol-gel process in the creation of organic/inorganic hybrid materials. The well-known two-step network-building sol-gel process consists of the hydrolysis of a metal alkoxide in the first stage and the polycondensation reaction in the second. Metal-organic alkoxides, in particular silica, are of special interest in this method because they can create an oxide network in organic matrices.

The sol-gel process of alkoxy silane can be explained as hydrolysis (Eq. (1)-(3)):



where, the alkyl group is denoted by R.

Eq. (4) shows the nature of totally condensed silica after the completion of the sol-gel process of reaction:



TEOS, which is easy to purify and has a somewhat slow reaction rate, is the most used ceramic precursor [52]. The kinetics of the hydrolysis and condensation reactions in the sol-gel process is influenced by a number of factors, including the silane and water ratio, temperature, the type of solvent, the catalyst, and others. The sol-gel methodology beats the traditional blending method because it can accurately regulate the shape or surface features of the developing inorganic phase in the polymer matrix by adjusting these reaction parameters.

To increase silicon's limited reactivity, catalysts that are acidic or basic are frequently utilized. It has been shown that basic catalysis frequently results in opaque composites with phase dimensions that are significantly larger than 100 nm and more typically in the micrometer range. It is impossible to classify these materials as nanocomposites. Alternately, when acid catalysis is used, transparent nanocomposites with unique morphologies and diameters below 100 nm are often produced. Therefore, the polymer/silica nanocomposites made by sol-gel techniques are commonly made using acid-catalysis [60, 61].

Two methods are typically used among the numerous other synthetic procedures that can be used in the sol-gel process to create polymer/silica hybrid materials, including:

(i) In situ creation of an inorganic network with an organic polymer that has already been produced [62]. It is necessary to determine the circumstances in which phase separation won't take place throughout the gel-forming and drying processes in order to produce optically clear materials. To lessen phase separation, covalent connections are frequently introduced between the inorganic and organic phases. The cosolvent that is utilized is the most crucial variable in regulating polymer solubility. THF, alcohol, and DMF are three regularly used solvents.

(ii) The stages of organic-inorganic synthesis can be differentiated based on the growth of organic and inorganic matter. Particle sizes and interactions between the dispersed and continuous phases frequently affect the characteristics of sol-gel nanocomposites. As previously discussed [54], hybrid materials can be broadly categorized into two fundamental types based on the nature of interfacial contact. Hydrogen bonds and van der Waals are examples of physical or weak phase interactions in C1 hybrids. Between the organic and inorganic phases of the hybrid in C2, there are powerful chemical connections [56].

3.3 In Situ Polymerization

3.3.1 General Polymerization

In situ polymerization has a variety of advantages. These include simple handling, quick processing, and enhanced performance of the finished items [63]. In situ polymerization typically consists of three sequential phases. The necessary surface modifiers are used to pretreat the nanoscale additives, and then the modified additives are disseminated into monomer (s). Bulk or solution polymerization then occurs. The nanocomposites are then created in the polymerization process. The dispersion and adhesion at the polymer and filler interfaces are without a doubt the most crucial elements that influence the characteristics of composites. When inorganic particles are premodified by a coupling agent, they may disperse uniformly in the polymer matrices [64]. Figure 3 shows the method of preparation of Epoxy / SiO₂ nanocomposites [65].

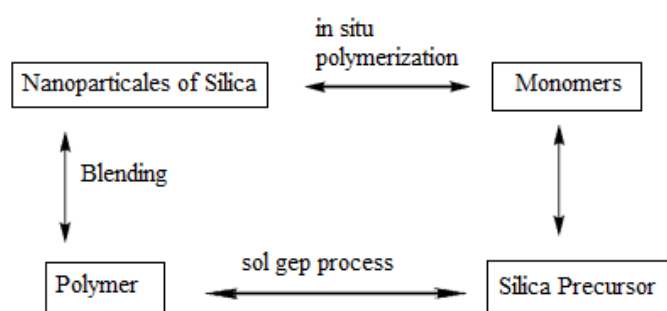


Fig. 3 preparation of Epoxy / SiO₂ nanocomposites

A process approximating bulk polymerization was used to polymerize the mixture at a high temperature in a nitrogen atmosphere after initial stirring to suspend the silica particles in the caproamide. The silica was pre-modified with aminobutyric acid before to polymerization. The experimental result showed that there is a uniform silicas dispersion takes place. The morphological study's findings demonstrated that the crystalline phase of these composites was unaffected by the presence of particles. The amount of filler and the manner in which it was dispersed all had an effect on the observed rise, which was clearly reinforced by the addition of filler. Both contain the yield point, identically.

It was observed that the latter parameters affected the results of compressive and tensile tests [66]. Literatures were observed to investigate the GPS treatment or APS treatment over 6/nano-SiO₂ with in situ polymerization [67]. TGA of silicas separated from the composites and an end group analysis of the composites demonstrated that functional silane treatment of nano-SiO₂ prior to in situ polymerizations of nylon 6 did not result in a noticeably different level of reactivity of silicas' surface groups. However, mechanical testing has shown that it may concurrently increase the composites' strength and hardness. The addition of a flexible layer to the interface was mostly responsible for this. To study how interphase affects nanocomposites, nano silica (nano-SiO₂) was prepared with coupling agents like aluminate, titanate, or silane. While the hydrogen-bonding connections along the metaphase may have helped the interfacial interactions

between the silica and the matrix for silicas treated with titanate or aluminate, the amino functional groups implemented could take part in the polymerization, giving rise in graft polymers on the silica surface [67].

Polyethylene terephthalate monomer mixed with organically modified silica nanoparticles was effectively used to create PET/silica nanocomposites in situ [68]. Results revealed that the nanoparticles were evenly distributed throughout the polymer matrix; their inclusion could hasten crystallization and raise the melting temperature, but it had no discernible impact on the synthesis process.

Two distinct types of polymer/silica nanocomposites have been produced by the free-radical polymerization of HEMA, either during the presence of HEMA-functionalized SiO₂ nanoparticles (T1) or with the concurrent in situ growth of the silica phase through the acid-catalyzed sol-gel polymerization of TEOS (T2). The particles in T1 systems had a traditional particle-matrix shape, although they preferred to group together. T2 systems had a finer morphology with a very open mass-fractal silicate structure that was thought to be molecularly bicontinuous with the organic phase [60].

Epoxy/silica is a well-researched nanocomposite system. As an organic matrix, epoxy resins offer great heat, moisture, and chemical resistance, as well as good adherence to a variety of substrates. They are unable to fully satisfy the demands of applications like epoxy molding compounds, nevertheless. They differ from inorganic materials in that they have low mechanical characteristics and a high CTE value. In order to reduce shrinkage during curing and CTE, enhance heat conductivity, and satisfy mechanical requirements, silica particles are frequently utilized as reinforcement in epoxy matrix. Typically, the hardener is introduced after the nano-silica, and epoxy polymer have been combined together to produce the curing reaction. Table 1 includes some examples of this type of in situ polymerized epoxy/SiO₂ nanocomposite, as well as a list of typical epoxy resins and hardeners for the production of such nanocomposites.

Table 1 Epoxy resin/ SiO₂ nanocomposites preparation by polymerization

Hardeners	SiO ₂	Literature reference
DDS + TGDDM	Dispersed in isopropanol (10-15 nm)	69
Polyamide-amine + DGEBA	---	70
SiO ₂ + DGEBA	Dispersed in MIBK (10-20 nm)	71
Z + DGEBA	Functionalized group particle, 400nm	72
DDS + bisphenol A	PAAM grafted (9 nm)	73
HMPA + DGEBA	100 nm	74
DDM, DEP + DGEBA, P Containing epoxy	Dispersed in MIBK (10-20 nm)	75
Aromatic hardener + DGEBA	15 nm	76, 77

3.3.2. Photopolymerization

UV light stimulates the polymerization process, enabling a quick conversion of the liquid monomer into a solid film with specialized physicochemical and mechanical properties. The UV light interacts with an appropriate photoinitiator to produce radical or cationic species, which trigger the curing reaction of reactive monomers and oligomers [69]. Because there are no solvents involved in the process, it is an environmentally friendly method. Energy is conserved because the substrate does not need to be heated as in conventional thermal curing. A related technique called electron beam (EB) driven polymerization technology has also drawn a lot of interest [70]. The formulation of UV-curable nanocomposites typically includes reactive dilutes (e.g., HDDA) and/or oligomers (e.g.: epoxy acrylate). In order to ascertain the relative importance of these two characteristics on the mechanical behavior, [71] created model-filled elastomers and varied the chemistry individually at the particle surface and the dispersion state. These samples were made in accordance with the method created by Ford et al. [72, 73], which involved photopolymerizing a concentrated dispersion of grafted silica particles in acrylate monomers. It has been demonstrated that using trialkoxysilane-modified nanosized silica in radiation-curable acrylate systems produces nanocomposite films that are more resistant to abrasion and scratching.

3.3.3 Surface-Initiated Polymerization

The establishment of unique interactions at the interface between organic and inorganic components is a crucial component in the creation of nanocomposite systems since the interface is largely responsible for the creation and characteristics of nanocomposites. Therefore, there is currently a lot of interest in the development of grafting techniques that can adjust the surface characteristics of mineral substrates. At the surface of the particles, linear polymer chains have been grafted using two typical

methods. The "grafting-to" technique is one method, and the "grafting-from" technique is the other. The surface can be improved by grafting polymers to inorganic particles like silica, however, nongrafted chains frequently contaminate the surface. Therefore, it is still difficult to distinguish between grafted and non-grafted chains. Strong resistance between grafted polymer chains also prohibits the attachment of further ones, which in turn restricts graft density.

The "grafting-from" method, also known as surface-initiated polymerization, for example, in situ polymerization with monomer expansion of polymer chains from encapsulated initiators on mineral surfaces, in turn, gives rise to so-called "polymer brushes" or "hairy nanoparticles," seems to be a very flexible and promising

method. There are a variety of other beginning procedures, such as free radical polymerization [74-80], which combines conventional radical polymerizations.

4. Conclusions

In the present paper, literature related to the study of high-performance polymer matrix nanocomposite is reviewed in detail. It is observed that a planetary ball mill is useful for highly abrasive material and also gives fine and uniform particle size. The insoluble and thermally unstable polymer can be processed through polymerization techniques. For the preparation of polymer composites with high nanotube loading, the in-situ process provides very good miscibility with almost any type of polymer. High chemical homogeneity can be achieved through the Sol-Gel technique, and it operates at low pressure and temperature. The sol-gel process can control the morphology and size of particles. Also, the sol-gel technique can produce organic and inorganic hybrids.

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