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Polymer Nanocomposite: An Overview

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ABSTRACT: Present article gives us the brief introduction of the polymer nanocomposites (PNCs), synthesis techniques, characterization tools and their applications in the various fields. In the field of nanotechnology, polymer based nanocomposites have become a great area of current research and development. PNCs also include other important areas including barrier properties, biomedical, opto-electronics applications and for fuel cell. Polymer based nanocomposite has advantages of low cost, high strength and wide spectrum. Also present review emphasizes on the techniques of synthesis and characterization of nanopolymer.

KEY WORDS: Polymer, nanocomposite, synthesis methods, characterization.

I. INTRODUCTION

In recent years, the researcher has achieved good progress in the grounding of a variety of PNCs and in understanding the basic features that determine their optical, thermal, mechanical, electrical and magnetic properties [1]. Nanocomposite refers to multiple materials in which at least one of the components has its dimension in the nanometre range. It has been known that the addition of nanofillers such as carbon black to rubbery polymers has a physically strong effect on the properties of these materials [2]. Also all nanomaterials which are under the process of investigation [3]. The use of inorganic nanoparticle into polymer matrix can give high performance of new material that finds application in several industries due to the fact that when different materials are joint to form various structures, the properties of the resultant materials depend on the constituent material [4]. PNC consist of a polymer or copolymer have nanoparticle or nanofiller diffuse in the polymer matrix. These may be of different shape (e.g. platelets, fibres, spheroids), but one dimension must be in the range of 1 to 50 nm. In the simplest case, adding of nanoparticles in polymer matrix can improve its performance, frequently in extremely dramatic degree, by just capitalizing on naturally. This approach is particularly efficient in yielding high performance composite, then good dispersion of the filler is achieved and the properties of the nanoscale filler are significantly different. Alternatively high performance nanocomposite consequences the composite properties based on the surface of the fillers, since nano particulates contain an extremely high surface area to volume ratios when good dispersion are achieved [5].

Composite is the system composed of two or more physically distinct phases whose combination produced aggregate properties that are different from those its constituents. Figure 1 shows the schematic illustration of two different types of thermodynamically achievable PNCs. If the filler has a nanometer width and a high aspect ratio (30-1000) plate-like structure, it is classified as a layered nanomaterials [6]. In general, nanomaterials provide reinforcing efficiency because of their high aspect [7]. The properties of a nanocomposite are due to the highest degree influenced by the dimension scale of its constituent phases and the quantity of mixing between the two or more phases. Nanocomposites are matrix material such as rubber, plastic polyolefin with a small content of nano scale material. Usually less than 5% of nanomaterials is used to improve thermal or mechanical properties. A typical way to produce nanocomposites are in-situ-polymerization and melt blending/compounding. These types of nanomaterials are commonly melt blended with plastics: nano clay, nanotube and nano scale particles (e.g. SiO₂, Ag, ZnO₃) [8].

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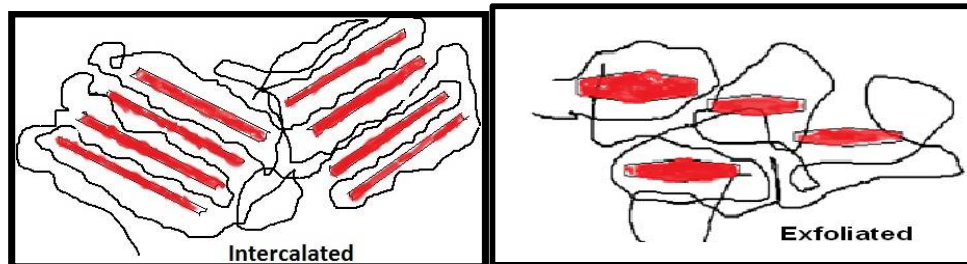


Figure1. Schematic illustration of two different types of thermodynamically achievable PNCs.

In a number of those areas, fundamental studies of mechanical, electrical, thermal, optical and chemical properties are required for applications. For manufacturing of nano-phased structural polymer composite material, the first step will be the fabrication method. Some of the widely used methods for manufacturing conventional composite parts are wet lay-up, pultrusion, resin transfer molding (RTM), vacuum assisted resin transfer molding (VARTM), autoclave processing, prepreg method, filament winding, fiber placement technology, etc. [9]. Wet lay-up is a easy method compared to other composite manufacturing methods; it allows the resin to be applied only in the die, but the mechanical properties of the product are poor due to voids and the final product is non-uniform. Pultrusion is a cheap continuous process with a high manufacture rate. But near the die assembly, the materials accumulate and can create a jam. The RTM is a closed die process [10]. In this process resin flood and fiber wet-out are serious issues; resin flows in the plane as well as in the sloping orders of the perform. The VARTM is an adaptation of the RTM; a widely used single-sided tooling procedure such as open molds are used to build the parts using vacuum [11]. This process has certain advantages like a relatively cheap for high volume production, very bulky and complex parts are possible with improved surface finish, higher fiber volume fraction than hand lay-up and as it is a closed system, it reduces environmental concerns more than hand lay-up. Both in RTM and VARTM, if the resin viscosity is high, it restricts the flow of resin. The effect of nanoconstituents on the matrix resins creates a problem in the altered resin viscosity and cure kinetics [12] and there is a possibility of dry spots or uneven distribution of resin over the entire volume of the reinforcement [13].

1.1 Classification of nanocomposites

As in the case of microcomposites, nanocomposite materials can also be classified, according to their matrix materials in three different categories such as Ceramic Matrix Nanocomposites (CMNC); Metal Matrix Nanocomposites (MMNC) and Polymer Matrix Nanocomposites (PMNC) [14]. Nanocomposite systems, including those reinforced with CNTs, have been widely studied since 1990s, there has been a stable and continuous increase in the number of publications on the subject, as well as reviews from time to time. In spite of this growth, the majority of the reviews describes the current status of only one type of nanocomposite. Thus, there are only two reviews on CMNC and three on CNT-reinforced nanocomposites and a quite large number on PMNC [15-16]. In the case of PMNC, reviews deal with processing aspects, including those on layered silicates conducting and biodegradable polymer-based systems, fibre reinforced and structure/morphology/property aspects as well as with applications and perspectives, including key opportunities and challenges in the development of structural and functional fibre nanocomposite [17]. Polymer materials are widely used in the industry due to their ease of production, lightweight and often ductile nature. However, they have some disadvantages such as low modulus and strength compared to metals and ceramics. In this context, a very effective approach to improve mechanical properties is to add fibres, whiskers, platelets or particles as reinforcements to the polymer matrix [18]. For example, polymers have been filled with several inorganic compounds, either synthetic or natural, in order to increase heat and impact resistance, flame retardancy and mechanical strength and to decrease electrical conductivity and gas permeability with respect to oxygen and water vapour. Furthermore, metal and ceramic reinforcements offer striking routes to certain unique magnetic, electronic, optical or catalytic properties coming from inorganic nanoparticles, which add to other polymer properties such as processability and film forming capability. Using this approach, polymers can be improved while keeping their lightweight and ductile nature. Another important aspect is that nanoscale reinforcements have an exceptional potential to generate new phenomena,

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which leads to special properties of these materials as will be seen later [19]. It may be pointed out that the reinforcing efficiency of these composites, even at low volume fractions is comparable to 40-50% of fibres in microcomposites. Addition of reinforcements to a wide variety of polymer resins produces a dramatic improvement in their biodegradability. This underlines a good example of PMNC as promising systems for ecofriendly applications [20].

II. MATERIALS AND METHODS

The following methods have been used for the synthesis of PNCs:

2.1 Template synthesis (sol-gel technology)

In this technique, the clay raw materials are synthesized in the polymer matrix, using an aqueous solution (or gel) containing the polymer and the silicate building blocks. During the process, the polymer adds the nucleation and growth of the inorganic host crystals and gets trapped inside the layers as they grow [21]. Theoretically, this method is possible by promoting the dispersion of the silicate layers in a one-step process, without the presence of the onium ion, it is a serious disadvantage. First of all, the synthesis of clay raw minerals generally requires high temperatures, which decompose the polymers. An exception is the synthesis of hectorite-type clay minerals in which needing can be performed under relatively calm conditions. The other problem is the aggregation tendency of the growing silicate layers. Although this technique is widely used for the synthesis of double-layer hydroxide-based nanocomposites. However, it should be mentioned that several workers have successfully applied it for the preparation of nanocomposite materials. For example, Carrado et al. and Xu synthesized hectorites from gels consisting of silica, magnesium hydroxide, lithium fluoride and polymers like poly(vinyl alcohol), polyaniline and polyacrylonitrile. Obviously, some silicate layers aggregated, but most of them remained uniformly distributed in the polymer matrix.

2.2 Intercalation of polymer or prepolymer from solution

In this technique, the layered silicate is exfoliated into the single layers using a solvent in which the polymer (or prepolymer in case of insoluble polymers, such as polyimide) is soluble. It is well known that such layered silicates, due to the weak forces that mound the layers together can be easily spread in an adequate solvent. After the organoclay has enlarged in the solvent, the polymer is added to the solution and intercalates between the clay layers. The final step consists of removing the solvent, either by vaporization, generally under vacuum, or by precipitation. Upon solvent removal the sheets reassemble, sandwiching the polymer to form a nanocomposite structure. Under this process are also gathered the nanocomposites obtained through emulsion polymerization where the layered silicate is spread in the aqueous phase. The main advantage of this method is that intercalated nanocomposites can be synthesized that are based on polymers with low or even no polarity. However, the solvent approach is difficult to apply in industry owing to problems associated with the use of large quantities of solvents [22,23].

The intercalation of a polymer from a solution is a two-phase process as shown in Figure 3 in which the polymer replaces an intercalated solvent. Such a replacement requires a negative variation in the Gibbs free energy. It is thought that the diminished entropy owed to the confinement of the polymer is rewarded by an increased due to desorption of intercalated solvent molecules. In other words, the entropy gained by desorption of solvent molecules is the heavy force for polymer intercalation from solution [24]. Even though this method has been mainly used with water soluble polymers, such as PEO, PVE, PVP and PAA [25], intercalation from non-aqueous solutions has also been reported [26]. For example, HDPE-based nanocomposites have been prepared by dissolving HDPE in a mixture of xylene and benzonitrile through dispersed OMLS. The nanocomposite was then recovered by precipitation from THF [27]. PS/OMLS exfoliated nanocomposites have also been prepared by the solution intercalation process by mixing pure PS and organophilic clay with adsorbed cetylpyridium chloride [28]. Similarly, several studies have focused on the preparation of PLA-layered silicate nanocomposites using intercalation from solution. The first attempts by Ogata [29], involved dissolving the polymer in hot chloroform in the presence of organomodified MMT. However, TEM and WAXD analysis revealed that only microcomposites were formed and that an intercalated morphology was not achieved. In a shortly study, Krikorian and Pochan [30] prepared PLA nanocomposites using dichloromethane as the polymer solvent and as the OMLS dispersion medium. The authors obtained intercalated or exfoliated nanocomposites, depending on the type of OMLS used, that is, exfoliated nanocomposites were formed when diols were present in the organic modifier of the clay, due to the favorable enthalpic interaction between these diols and the CO bonds in the PLA backbone [31] reported the preparation of PLA-based nanocomposites with different kinds of

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OMLS via solution intercalation using N,N-dimethylacetamide (DMA). In the case of polymeric materials that are infusible and insoluble even in organic solvents, the only possible route to produce nanocomposites with this method is to use polymeric precursors that can be intercalated in the layered silicate and then thermally or chemically converted into the desired polymer [32]. Summarizing the above, a number of nanocomposites have been produced by intercalation from a solution, it is important to note that, in using this technique, intercalation only occurs for certain polymer/clay/solvent systems, meaning that for a given polymer one has to find the right clay, organic modifier and solvents [33].

2.3 Melt intercalation

This technique consists of blending the layered silicate with the polymer matrix in the molten state. Under such conditions if the layer surfaces are suitably well-matched with the chosen polymer, the polymer can move slowly into the interlayer space and form either an intercalated or an exfoliated nanocomposite [34]. Among the above mentioned methods, in situ polymerization and melt intercalation are considered as commercially attractive approaches for preparing polymer/clay nanocomposites. Melt intercalation is especially of practical interest, since it presents significant advantages that will be discussed.

2.4 Ex situ processes

Ex situ processes can be synthesized in an external synthesis step, are added or mixed with monomer or resin (organic solution), usually followed by a polymerization. In more elaborated setup the nanomaterials were first surface functionalized and then added to organic solution. Other approaches used commercial nanoparticle, applied coupling agents and finally blended the particle with polymer powder. This method leads to bulk composite material [35]. The Ex situ process for the formation of nanocomposites of polystyrene-nano graphite is shown in Figure 2.

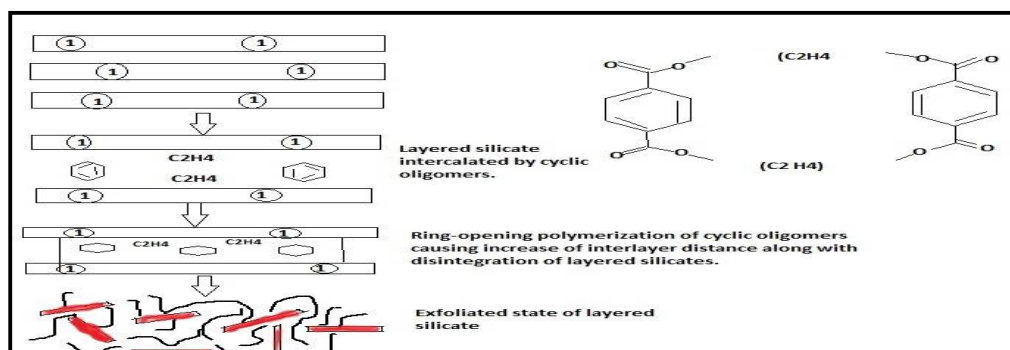


Figure 2. Schematic illustration of the Ex situ process for the formation of nanocomposites of polystyrene-nano graphite.

2.5 Chemical in situ method

This method mainly uses a chemical reaction in a liquid environment to make a nanocomposite. In this nanoparticles are synthesized in the first step, mostly as a sol or dispersed in a solution, followed by a second step where a monomer or resin is added and finally brought to polymerization [36].

2.6 Physical in situ method

Physical method is generally gas-phase method. They are able to synthesize in-situ functionalized or encapsulated nanoparticle. Their common feature is that relate energy to transform chemical compound (precursor and gas) into inorganic nanoparticle and by a successive covering step organic compound is grafted on the nanoparticles [37].

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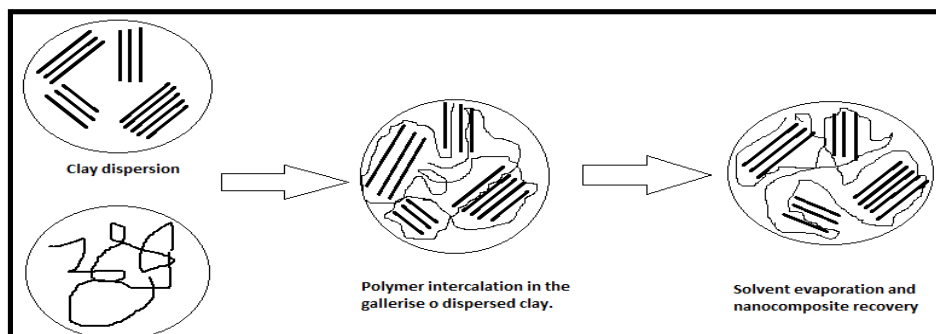


Figure 3. Schematic representation of PLS obtained by intercalation from solution.

III. CHARACTERIZATION TECHNIQUES

Characterization tools are crucial to understand the basic physical and chemical properties of PNCs. For structural applications, it facilitates the study of emerging materials by giving information on some intrinsic properties [38]. Various techniques for characterization have been used extensively in PNC research. The commonly used powerful techniques are wide-angle X-ray diffraction (WAXD), small-angle X-ray scattering (SAXS), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) [39]. The SEM provides images of outside features connected to the sample. However, there are two other microscopes, scanning probe microscopy (SPM) and scanning tunneling microscopy (STM), which are essential in nanotube research. The SPM uses the interaction between a sharp tip and a surface to obtain an image. In STM, a sharp conducting tip is held suitably close to a surface (typically about 0.5 nm), such that electrons can tunnel across the gap [40]. This system provides a surface structural and electronic information at the atomic level. The invention of the STM inspired the development of other SPM, such as the atomic force microscope (AFM). Raman spectroscopy has also proved a useful study of carbon-based material properties [41]. Due to the easiness and availability, WAXD is the most commonly used to explore the nanocomposite structure and rarely to study the kinetics of the polymer melt intercalation. In layered silicate nanocomposite systems, a fully exfoliated system is characterized by the absence of intensity peaks in WAXD pattern, which corresponds to d-spacing of at least 6 nm. Therefore, a WAXD pattern about the mechanism of nanocomposite formation and their structure are tentative issues for making any conclusion. On the other hand, TEM allows a qualitative understanding of the internal structure, spatial distribution of the various phases and views of the defective structure through direct visualization, in some cases of individual atoms. Therefore, TEM complements WAXD data. Small-angle X-ray scattering (SAXS) is typically used to observe structures on the order of 10\AA or larger. The TEM, AFM, and SEM, are also required to characterize nanoparticle, carbon nanofiber dispersion, or distribution. However, X-ray diffraction has found relatively limited success in CNT research. For thermal characterization and to study the core behavior PNCs, the commonly used techniques are differential scanning calorimeter (DSC), thermogravimetric analysis (TGA), thermomechanical analysis (TMA), Fourier-transform infrared (FTIR), dynamic modulus analysis (DMA), rheometer, etc.

IV. MECHANICAL PROPERTIES

4.1 The reinforcing mechanism of layered silicates

The first mechanism that has been put forward to explain the reinforcing action of layered silicates is one also valid for conventional reinforcements, such as fibers, which is schematically depicted in Figure 5. That is, rigid fillers are naturally resistant to straining due to their high moduli. Therefore, when a relatively softer matrix is reinforced with such fillers, the polymer, particularly that adjacent to the filler particles, becomes highly controlled mechanical. This enables a significant portion of an applied load to be carried by the filler, assuming, of course, that the bonding between the two phases is adequate [42]. From this mechanism, it becomes obvious that the superior the surface of the filler in contact with the polymer, the greater will be the reinforcing effect. This could somewhat explain

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why layered silicates, having a very high specific surface area, (on the order of $800\text{m}^2/\text{g}$) dramatic improvements of modulus even when present in very small amounts in a polymer. In fact, the low silicate loading required in nanocomposites to effect significant property improvements, is probably their most distinguishing characteristic. In most conventionally filled polymer systems, the modulus increases linearly with the filler volume fraction, whereas for nanocomposites much lower filler concentration increase the modulus sharply and to a much larger extent.

4.2 Modulus and strength

In general, the addition of an purely modified layered silicate in a polymer matrix result in significant improvements of Young's modulus, for a number of different materials. For example, Gorrasi et al. [43] reported an increase from 216 to 390 MPa for a PCL nanocomposite containing 10 wt.% ammonium-treated montmorillonite, while in one more study, Young's modulus was increased from 120 to 445 MPa through the addition of 8 wt.% ammonium treated clay in PCL. Similarly, in the case of nylon 6 nanocomposites obtained during the intercalative ring opening polymerization of caprolactam, a large increase in the Young's modulus at quite low filler content has been reported.

4.3 Toughness and strain

The brittle behavior frequently exhibited by nanocomposites probably originates from the formation of microvoids due to debonding of clay platelets from the polymer matrix upon failure. This has been testified throughout careful inspection of fracture surfaces and is also linked with observations by in situ deformation experiments using TEM [44].

4.4 Heat distortion temperature

Heat distortion temperature or heat deflection temperature (HDT) is the temperature at which a polymer deforms below a specified load. Hence, it is an index of a polymeric material's heat resistance towards applied load and is assessed by the procedure given in ASTM D-648. In general, improvements of HDT are reported by nanocomposite formation. Usually, a significant increase is achieved for clay contents of approximately 5 wt.% and then HDT values level off for higher clay loadings. For example, Kojima et al [45] shown that the HDT of pure PA6 increases up to 90°C after nanocomposite preparation with OMLS and also they have reported the clay content dependence of HDT of PA6/MMT nanocomposites. More specifically, they shown that there is a marked increase in HDT from 65°C for neat PA6 to 152°C for 4.7 wt.% nanocomposite. Beyond that MMT loading, the HDT of the nanocomposites levels off. Moreover, the HDT values of various PA6 nanocomposites prepared with clay lamellae of different lengths shown that the HDT also depends on the aspect ratio of dispersed clay particles.

4.5 Optical clarity

Microsized particles used as reinforcing agents scatter light, thus reducing light transmittance and optical clarity. On the other hand, layered silicate platelets, their micron lateral size, are just 1 nm thick. Thus, when single layers are dispersed in a polymer matrix, the resulting nanocomposite is optically clear in the visible region, whereas, there is a loss of intensity in the UV region (for $< 250\text{ nm}$) mostly due to scattering by the MMT particles. There is no marked decrease in the clarity due to the nanodispersed fillers. This is a general behavior in UV/vis transmittance for thick films (3-5 mm) of polymer/MMT nanocomposites based on PVA, PP and several epoxies [46].

V. NANOCOMPOSITES: ADVANTAGES AND APPLICATIONS

PNC materials frequently exhibit properties superior to conventional composites, such as strength, stiffness, thermal and oxidative stability, barrier properties, as well as flame retardant behavior. These improved properties are usually attained at lower filler content in comparison with conventionally filled systems. Therefore, polymer-layered silicate nanocomposites are future lighter in weight than a conventional composite, which makes them quite reasonable for specific applications. Moreover, for systems with favorable thermodynamics of mixing, the organoclay can be incorporated in the final stages of polymer processing (e.g. extrusion, injection or compression molding) to obtain nanocomposite materials. Thus, polymer nanocomposites are amenable to most of the common processing techniques in today's industrial practice which will lower the barriers to their commercialization [47-48]. Another unique aspect of nanocomposites is the lack of belonging trade-offs. Traditionally, blend or composite formulations require trade-offs

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between desired act, mechanical properties, cost and processibility. However, polymer nanocomposite technology provides a route around these traditional limitations and offers, for the first time, the opportunity to design materials missing the compromises typically found in conventionally filled polymers. The above mentioned attractive characteristics of polymer-layered silicate nanocomposites already suggest a variety of possible industrial applications: automotive (gas tanks, bumpers, interior and exterior panels), construction (building sections and structural panels), aerospace (flame retardant panels and high performance components), food packaging, textiles, etc. It is the reason that many companies have taken a strong interest and have invested in developing nanoclays and polymer nanocomposites. Among these, the first commercial product of clay-based polymer nanocomposites was the timing-belt cover made from PA6 nanocomposites by Toyota Motors in the early 1990s. This timing-belt cover exhibited good rigidity, excellent thermal stability and no warp. It also saved the weight up to 25%. Later, General Motors and partners Basell, Southern Clay Products and Blackhawk Automotive Plastics announced external automotive body parts (step-assist) made from thermoplastic, olefin layered silicate nanocomposites. A TPO nanocomposite with as little as 2.5% layered silicate is as stiff and much lighter than parts with 10 times. Also quite interesting are potential applications of nanocomposites based on biodegradable polymers. Such polymers have become indispensable in a wide range of applications. However, despite their attractive degradation characteristics and significant demand for such materials, the lack of structural and functional stability prevents currently available biodegradable polymers from having widespread commercial impact [49-52]. Therefore, biodegradable polymer-based nanocomposites show to have a very bright future for a wide range of applications as high-performance biodegradable materials. Layered silicate nanoparticles, when dispersed within the matrix of a fiber-reinforced polymer (FRP), can delay the diffusion of environmental moisture and other chemicals to the fiber-matrix interface where their presence can result in delamination and fiber weakening. The possible growth paths for nanocomposites applications is shown in Figure 4.

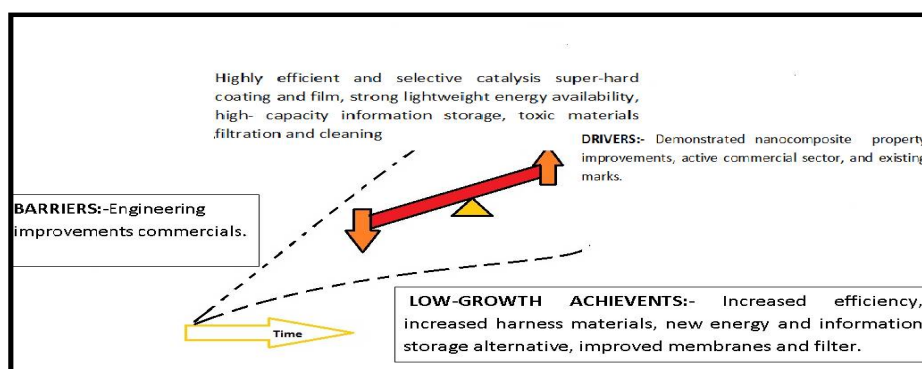


Figure 4. Possible growth paths for nanocomposites applications.

VI. CONCLUSION

Polymers are generally insulators but PNCs have good conductivity, stability and having good sensing properties. The research created on PNCs is extremely broad, encompassing areas such as electronics, computing, data storage, communications, aerospace, health, medicine, energy, environmental, transportation and defence. The focus of this review is to highlight the state of knowledge in processing, manufacturing, characterization, material properties, challenges and the potential applications for the most common PNCs such as automotive and textile. It is concluded that PNCs are easy to synthesize and the laboratory model based on polymeric materials is better for fabrications of various devices.

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