UNIVERSITI PUTRA MALAYSIA

OPTICAL, STRUCTURAL, THERMAL AND PHYSICAL PROPERTIES OF POLYMETHYLMETHACRYLATE AND POLYSULFONE NANOCOMPOSITES

RAOUF MAHMOOD RAOUF

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OPTICAL, STRUCTURAL, THERMAL AND PHYSICAL PROPERTIES OF POLYMETHYLMETHACRYLATE AND POLYSULFONE NANOCOMPOSITES

By

RAOUF MAHMOOD RAOUF

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

August 2016

DEDICATIONS

To the spirit of my father Immaculate……

My mother, Allah saves her……

To my dear faithful wife

My children.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

OPTICAL, STRUCTURAL, THERMAL AND PHYSICAL PROPERTIES OF POLYMETHYLMETHACRYLATE AND POLYSULFONE NANOCOMPOSITES

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August 2016

Chairman : Associate Professor Zaidan Abdul Wahab, PhD
Faculty : Science

Transparent polymers, such as polymethylmethacrylate (PMMA) and polysulfone (PSF), contain absorbing chromophores as a part of their structure. The chromophoric groups are able to absorb UV energy and involved in the photochemical degradation reactions leading to the formation of hydroperoxides and chain scission. Superb properties of environmentally friendly cellulose esters encourage us to use them as an inhibitor for photochemical interaction in addition to the high-formation ability. Moreover, the absorption peak of indium oxide nanoparticles (nano-In_2O_3) at round 280 nm. contribute to curb the photochemical interaction in the polymer matrix. The present thesis aims to improve PMMA and PSF in order to protect themselves and covered surfaces from the impact of UV radiation by using three environmentally friendly cellulose esters and the nano-In2O3. Two sets of transparent nano-composites based on PMMA and PSF were prepared separately with cellulose acetate butyrate (CAB), cellulose acetate propionate (CAP) and cellulose acetate phthalate (CAT) added with nano-In2O3 using a twin screw extruder in various percentage concentrations.

The preparation process was divided into two stages; the first was preparing transparent samples from PMMA and PSF separately with CAB, CAP and CAT. The second was adding nano-In_2O_3 to selected blend concentrations. Scans over UV and visible spectra for all highly transparent samples were made from 220 nm. to 800 nm. using a spectrophotometer. The results showed that the absorbance peak in the ultraviolet region for pure PMMA at 226 nm and for pure PSF at 268 nm, whilst the transmittance peak in the visible range for pure PMMA at 798 nm and for PSF at 712 nm. The results also showed that the increase in CAB, CAP and CAT percentage concentrations in blends reduce UV rays' absorbance while maintaining a high transmittance. The selection of specific concentrations PMMA/10%CAB, PMMA/10%CAP, PMMA/1%CAT, PSF/0.2%CAB, PSF/0.2% CAP and PSF/0.1% CAT represents less absorbance value within UV damage threshold for PMMA and PSF. The effect of nano-In_2O_3 percentage concentrations on the absorbance and transmittance spectrum for PMMA and PSF was done using a spectrophotometer.

The results demonstrated that PMMA/0.05%/nano-In_2O_3 and PSF/0.02%/nano-In_2O_3 have maximum UV absorbance with high transmittance. Morphological and structural characterizations of selected samples were studied by means of Scanning Electron Microscopy (SEM) and Differential Scanning Calorimetry (DSC). The SEM results showed single phase for PMMA blends and composite surfaces, while PSF blends surfaces showed a size reduction in all blend and composite samples. The DSC results indicated that all samples are miscible. The thermogravimetry analysis (TGA) was used to characterize thermal behavior of the samples. The results indicated that pure PMMA degraded in three steps while pure PSF degraded in two steps. The TGA analyses also indicated that all CAB, CAP and CAT blends with PMMA and PSF have good thermal stability and adding nano-In2O3 maintained the thermal stability for all samples. The dynamic mechanical Analysis (DMA) showed that the storage modulus and loss modulus of PMMA significantly increased by the incorporation of nano-In2O3. Finally, the samples PMMA/10%CAB/0.05% In_2O_3 and PSF/0.2%CAP/0.02% In_2O_3 are the best overall properties in this work.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

SIFAT OPTIK, STRUKTUR, TERMA DAN FIZIKAL POLYMETHYLMETHACRYLATE DAN POLYSULFONE

Oleh

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Dua set komposit Nano lutsinar, yang berasaskan Polimetilmetakrilat (PMMA) dan polisulfon (PSF), mengandungi chromophores sebagai sebahagian daripada struktur mereka. Kumpulan-kumpulan chromophoric dapat menyerap tenaga UV dan terlibat dalam tindak balas fotokimia degradasi yang membawa kepada pembentukan hydroperoxides dan rantaian scission. Ciri-ciri luar biasa yang dimiliki oleh ester selulosa yang turut bersifat mesra alam menggalakkan kita untuk menggunakannya sebagai perencat bagi interaksi fotokimia sebagai tambahan kepada keupayaan-pembentukan tinggi. Selain itu, puncak penyerapan nanopartikel indium oksida (nano In_2O_3) pada pusingan 280 nm. membendung interaksi fotokimia dalam matriks polimer. Kajian bertujuan untuk meningkatkan PMMA dan HPG sebagai langkah melindungi diri mereka dan permukaan dilindungi daripada kesan sinaran UV dengan menggunakan tiga ester selulosa mesra alam dan nano-In_2O_3. Dua set komposit Nano lutsinar berdasarkan PMMA dan PSF telah disediakan secara bebas dengan selulosa asetat butyrate (CAB), selulosa asetat propionat (CAP) dan selulosa asetat phthalate (CAT) ditambah dengan nano In_2O_3 menggunakan penitis skru berkembar dalam pelbagai peratusan kepekatan.

Proses penyediaan telah dibahagikan kepada dua peringkat; peringkat yang pertama ialah penyediaan sampel telus dari PMMA dan PSF berasingan dengan CAB, CAP dan CAT. Peringkat yang kedua ialah menambah nano In_2O_3 kepada kepekatan campuran yang dipilih. Imbasan lebih UV dan spektrum yang boleh dilihat untuk semua sampel sangat telus dibuat daripada 220 nm. hingga 800 nm menggunakan spektrofotometer. Hasil kajian menunjukkan bahawa puncak keserapan di rantau ultraviolet untuk PMMA tulen adalah pada 226 nm. dan untuk PSF tulen adalah pada 268 nm, manakala puncak pemindahan dalam julat yang boleh dilihat untuk PMMA tulen ialah pada 798 nm dan untuk PSF ialah pada 712 nm. Keputusan juga menunjukkan bahawa peningkatan dalam kepekatan CAB, CAP dan peratusan CAT dalam campuran mengurangkan kuantiti sinar UV ' walaupun mengekalkan kadar pemindahan yang tinggi. Pemilihan kepekatan tertentu PMMA / 10% CAB, PMMA / 10% CAP, PMMA / 1% CAT, PSF /% CAB 0.2, PSF / 0.2% CAP dan PSF / 0.1%

CAT mewakili kurang nilai kuantiti dalam kerosakan UV ambang untuk PMMA dan PSF. Kesan kepekatan peratusan nano In_2O_3 pada kuantiti dan pemindahan spektrum untuk PMMA dan PSF telah dilakukan dengan menggunakan spektrofotometer. Keputusan menunjukkan bahawa PMMA / 0.05% / nano In_2O_3 dan PSF / 0.02% / nano In_2O_3 mempunyai kuantiti UV maksimum dengan kadar pemindahan yang tinggi. Sampel akhir telah disediakan dari semua kepekatan. Morphological yang dipilih dan pencirian struktur sampel yang dipilih telah dikaji dengan cara menggunakan Scanning Electron Microscopy (SEM) dan Differential Scanning Calorimetry (DSC). Keputusan SEM menunjukkan fasa tunggal untuk campuran PMMA dan permukaan komposit, manakala PSF telah menggabungkan permukaan menunjukkan pengurangan saiz dalam semua gabungan dan sampel komposit. Keputusan DSC menunjukkan bahawa semua sampel terlarut boleh bercampur. Analisis termogravimetri (TGA) telah digunakan untuk mencirikan kelakuan terma sampel. Keputusan analisis menunjukkan bahawa PMMA tulen berkurangan dalam tiga langkah sementara PSF tulen berkurangan dalam dua langkah. TGA analisis juga menunjukkan bahawa semua CAB, CAP dan CAT campuran dengan PMMA dan PSF mempunyai kestabilan haba yang baik, terutamanya dengan menambah nano In2O3 bagi mengekalkan kestabilan haba untuk semua sampel. Mekanikal Analisis dinamik (DMA) menunjukkan bahawa penyimpanan modulus dan kehilangan modulus PMMA meningkat dengan ketara. Secara keseluruhannya, sampel PMMA/10%CAB/0.05% In_2O_3 dan PSF/0.2%CAP/0.02% In_2O_3 meuunjukkan sifat uvggul dalam projek ini.

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LIST OF ABBREVIATIONS

AFM	Atomic force microscopy
ATR-RTIR	Attenuated total reflection-Fourier transform infrared spectroscopy
BAM	Brewster angle microscopy
CCMC	Carbon-matrix composites
CAB	Cellulose acetate butyrate
CAT	Cellulose acetate phthalate
CAP	Cellulose acetate propionate
CMC	Ceramic-matrix composites
CDS	Chemical solution decomposition
DSC	Differential scanning calorimetry
DTA	Differential thermal analysis
DMF	dimethylformamide
DLS	Dynamic light scattering
DMA	Dynamic mechanical analysis
DMTA	Dynamic mechanical thermal analysis
LCR	Electronic test equipment
FESEM	field emission scanning electron microscopy analyses
FTIR	Fourier Transform Infrared Spectroscopy
GPC	Gel permeation chromatography
HRTEM	High resolution transmission electron microscopy
MMC	Metal-matrix composites
MW	Mo1lecular weight
NCP	Nanocomposites
NM	Nanomaterials
NPS	Nanoparticles
NIR	Near infrared
NMR	Nuclear magnetic resonance spectroscopy
OLS	Okazaki large spectrograph
OMC	Organic-matrix composites

PL	Photoluminescence
PC	Polycarbonate
PDLC	Polymer dispersed liquid crystals
PNC	Polymer nanocomposites
PMCs	Polymer-matrix composites
PMMA	Polymethylmethacrylate
PSF	Polysulfone
RAFT	Reversible addition fragmentation chain transfer
SEM	Scanning electron microscope
STEM	Scanning transmission electron microscope
SEC	Size exclusion chromatography
SCT	Solution cast technique
SCM	Spin coating method
TG-DTA	Thermogravimetric differential thermal analysis
XRF	X-ray fluorescence
UVC	Ultraviolet range (100-280)
UV-VIS-IR	Ultraviolet-visible-near infrared spectrophotometer
WXRD	Wide-angle X-ray diffraction

LIST OF SYMBOLS

A	Absorbance of the same absorbed beam
Ca	Capillary number
ΔH_m	Enthalpy change during melting
ΔS_m	Entropy change during melting
Tg	Glass Transition Temperature
$\propto_e$	Molar absorption coefficient.
X_i	Number of molecules
I	The absorbed intensity of the same beam
θ	The angle between incident beam and planes
R	The droplet radius
ΔC_p	The heat capacity
λ	The incident beam wavelength
$I_\circ$	The beem intensity of special wavelength
τ	The ratio of the shear stress
C	The sample concentration
$\dot{\gamma}$	The shear rate
γ	The surface tension
w	The weight fraction
ρ	Viscosity ratio
η_d	Viscosity for the dispersed phase
η_m	Viscosity of the matrix
$\bar{M}_w$	Weight average molecular weight

CHAPTER 1

INTRODUCTION

1.1 Transparent Polymers

In a world full of different types of polymers it is perhaps the transparent ones that give the greatest benefit to our lives because it is involved in numerous applications (from eye glasses to solar concentrating lenses for spacecraft power and propulsion systems) (Edwards et al., 2000). It has now become indispensable in all areas of life. The structure of transparent polymers does not differ significantly from non-transparent (opaque) polymers, that only transparent polymers are often amorphous and the blends mostly miscible. There are about 120 types of transparent polymers most of them are acrylic with transmittance more than 90% and refractive index of 1.5-1.6. Polysulfone as one of transparent polymers that has a refractive index 1.63 and transmittance of about 80% (Seymour and Carraher, 1984). The most common transparent polymer is PMMA and polycarbonate (PC). In order to apply polymers in optical uses, they must have different refractive indices (Prasad, 2012). As a result of their functional performance polymers transparency is affected by UV radiation and weather conditions leading to a decrease in transparency (photolysis) because of oxidative degradation that lead to molecular chain scission (Hamdy et al, 2016; Sun et al., 2016).

1.2 Thermal Behavior of Polymers

The thermal transitions in polymers are slightly different and more complex than non-organic. First, the molecules are large enough to be suitably called polymers and do not exist in the gaseous state and they decompose rather than boil. Second, polymer consists of a mixture of molecules possessing different chain lengths (different molecular weights), so the transition from solid to liquid form of a polymer is rather diffuse and occurs over a temperature range between 2 and 10 °C depends on the polydispersity of the polymer. Third, the polymers become very viscous fluids (viscoelastic form) in the melting.

The molecular motion in a polymer is promoted by its thermal energy and opposed by the cohesive forces between structural pieces along the chain in addition to neighboring chains. These cohesive forces and, as a result, thermal transitions in polymers rely on polymer structure. In this regard, Polymeric materials are characterized by two major types of transition temperatures, the glass transition temperature (Tg) and the crystalline melting temperature (Tm).

The transition from the hard (brittle glass) to a softer (rubbery) state in amorphous polymers occurs through a temperature range called the glass transition temperature (Tg) and in the case of a partially crystalline polymer the transformation occurs only in the amorphous regions whilst the crystalline zones remain unchanged and act as reinforcing elements so the polymer be hard and tough. If heating is continued, the polymer crystalline zones initiate melting. The equilibrium crystalline melting point

(Tm) for polymers will be the temperature at which the last crystalline starts melting. It is noteworthy that the value of (Tm) depends on the degree of crystallinity and size distribution of crystallites (Ebewele, 2000; Odian, 2004).

1.2.1 The Glass Transition Temperature (Tg)

The glass transition temperature term observes in linear amorphous polymers, such as poly methylmethacrylate or polystyrene. It occurs at well-known temperatures. When the bulk material is heated to temperatures above the glass transition temperature (Tg) turns from brittle and glassy to soften like rubber or liquefy depending on their degree of cross-linking and/or molecular weight. In the case of semi crystalline materials (polypropylene, polyethylene) Tg is much lower than melting point and represents the temperature range in which on cooling the brittleness increases noticeably (Rieger, 2001).

Many physical properties like mechanical damping, heat capacity, coefficient of thermal expansion, electrical properties and refractive index change profoundly at Tg because they depend on the relative degree of freedom of molecular motion within polymeric materials and each property can be used to monitor the point at which Tg occurs (Nicholson, 2012; Rieger, 2001). The rheological definition of glass transition is that the glass transition occurs when the viscosity is 10^{12} Pa.s.

The Tg depends on the cooling rate because glass transition is kinetic property. When the cooling rate (normal cooling rate in glass studies 10K/min) increases, glass transition would occur at a high temperature; when the cooling rate decreases, glass transition would occur at low temperature (Zhang, 2008).

The most common methods for determining the glass transition temperature are Differential Thermal Analysis (DTA), Differential Scanning Calorimetry (DSC) and Dynamic Mechanical Analysis (DMA) (Rieger, 2001).

1.2.2 The Crystalline Melting Point (Tm)

Melting includes a change from the crystalline solid state to the liquid form. For low molecular weight materials, melting represents a first order thermodynamic transition characterized by discontinuities in the primary thermodynamic factors of the material system such as specific volume (density), heat capacity, transparency and refractive index. Melting happens when the change in free energy of the process is zero.

The crystalline polymers do not have clear melting points. This is because they are effectively mixtures, including components from a range of relative molar masses, each component of which melt at different temperature. Thus, low relative molar mass homopolymers melt at lower temperatures than high relative molar mass species. The size of the crystallites also influences the range of the melting temperature, as smaller and less perfect crystallites melt before large ones when the temperatures are heating up.

There are two other factors which influence the value of the temperature range over which melting takes place:

- First is the presence of co-monomer in the polymer, involving copolymerization, this factor reducing the melting point by shortening the crystallisable series length within the individual polymer molecules.

- Second is the presence of solvent trace or solvent plasticizer in the polymer, this factor dose so by increasing the relative mobility of the polymer molecules in the material, so reducing the necessary energy to take them into the liquid phase (Nicholson, 2012).

1.3 Polymer melt

Polymers can be illustrious by their properties, especially the change that occurs after heating, which relates to their change in stiffness and damping. Each polymer has individual physical properties that will set their use in practice. It is important to be able to determine the nature of an unknown polymer for many reasons. The first is to be able to determine the quality and dependability of a manufacturing process, to see that the final product is of a steady composition with time. The second is to match up polymers from different origins, of apparently similar groups. Finally, it is often important to be able to determine the nature of a plastic of unknown origin (Faria et al., 2007).

The melt blending is a complex procedure which includes melting of pellets, dispersive mixing, distributive mixing, and droplet cohesion. Dispersive mixing is a procedure in which the size of a dispersed phase decreases because of applied stresses, even as distributive mixing includes homogenization of the dispersed phase inside a matrix. Figure 1.2 shows how dispersed phase morphology creates throughout melt compounding. At first, pellets of the minor component melt and a thin film of the dispersed phase is structured on the surfaces of the mixing equipment because of shear forces. This film is precarious and creates holes as a consequence of interfacial unsteadiness, structuring a lace-like formation of thin filaments which at last separation into droplets. Without a compatibilizer at the phase interface a coarser morphology is formed because of droplets cohesion of, whilst a better dispersion is gotten with a compatibilizer (Cheerarot, 2012; Ryan, 1998; Scott and Macosko, 1995).

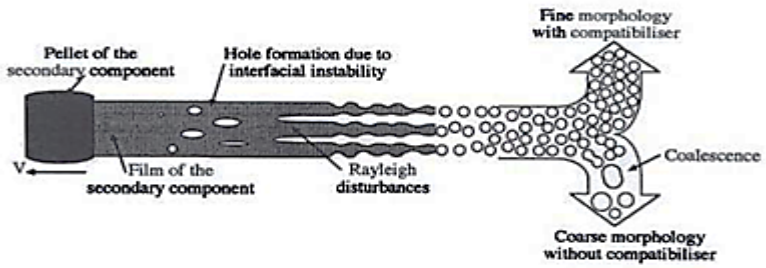

Figure 1.1: Diagram depicting morphology progress of the dispersed phases through melt mixing of immiscible polymers (Ryan, 1998).

1.4 Polymer Blends and Copolymers

1.4.1 Polymer Blends

Polymer blends are physical mixtures of two or more polymers (these polymers could be homopolymers or copolymers). It has been estimated that ~ 30% of all polymers are solid in mixed form. Attention on these materials has been increased firstly from the ability to develop mechanical properties. The phase behavior of polymer blend systems is a function of temperature, pressure and concentration. The huge majority of polymer blends has been found to be multiphase - either completely immiscible or multiphase with some limited mixing. This is mostly the result of the small combinatorial entropy of blending for the disparate polymer chains. The remaining part is a single phase, melt-miscible mixtures (Cheng, 2002; Ibrahim and Kadum, 2010).

Polymer blends consist of 30 – 40% of polymer products, and gradually more employed in the new materials design. Among the reasons that led to blends to be of considerable interest are:

• Adaptability in detail end product properties.

• Synergistic arrangements of blend ingredients to optimize the properties, such as improving mechanical, optical, thermal and electrical properties.

• Low investment cost of novel materials design and low cost.

Most profitable blends consist of two polymers and a small amount of a third compatibilizing polymer (typically copolymer). Polymer blends are combination of independent molecular distributions, which can vary in molecular weight MW and structure of functional groups.

Several techniques have been utilized to distinguish single and multi-phase character in polymer blends. Optical clarity is frequently used as a first indication, in the absence of crystallinity; a single-phase material is expected to be optically clear while a multi-phase mixture should be opaque. Optical and electron microscopy techniques are used often in an attempt to find out phase structure in polymer blends and block copolymers. The dependence on the atomic force microscopy (AFM) will help to characterize the phase-separated structure of phase separated blends and copolymers. Measurement of the location and number of Tg by thermal analysis techniques is the most common approach to evaluate phase behavior in a particular temperature range in polymer blends. In a binary mixture, if two Tg's are observed at the same temperatures as those of the component polymers, means that the polymers are completely immiscible. A single Tg between the components indicates miscibility. The intervals of Tg in miscible polymers are frequently broadened compared to those of the components, as a result of a distribution of local surroundings. There is also two Tg's but shifted inwards from the components. This is generally taken to indicate multiphase behavior with some mixing between the components. A partially miscible blend as well can happen in polymer blends (Cheng, 2002; Ibrahim and Kadum, 2010).

Many expressions have been used to predict Tg of miscible mixtures. The Fox-Flory equation, primarily derived for random copolymers, is probably the most widely used (Cheng, 2002; Ibrahim and Kadum, 2010).

Attempts have also been made to connect the experimentally derived (Tg)s of miscible blends to intermolecular interactions (Ibrahim and Kadum, 2010; Kwei et al., 1987; Lu and Weiss, 1992).

1.4.2 Copolymers

Copolymers are macromolecules that are created from two or more different monomer units. The basic arrangements of the building blocks are determined by relative monomer reactivities and the synthetic procedures used. Different chemical units can be arranged in random, blocky or alternating fashion. The most common block copolymers are: diblocks (where relatively long segments of monomers A and B are connected at one end); triblock copolymers (the An-Bin-An type); and multiblock copolymers ([A-A . . . . . A-B-B . . . . . B]x). The alternating and random copolymers frequently show a single phase structure, conversely high molecular weight (block and graft copolymers) often exhibits phase separated morphology. However, microphase separation can watch, because of the interconnectivity of the kinds, as opposed to separation of large scale, that found in the great bulk of polymer blends.

Thermoplastic elastomers are an important class of materials that are frequently derived from tri or multi-block arrangements. These polymers are consisting of a comparatively high concentration of soft (molecule) (blocks that are amorphous and have low Tg*)* besides two or more hard (molecule). The hard molecules provide physical crosslinks (reinforcement) and are either amorphous with Tg > room temperature or crystalline with a melting point above room temperature (Styrene-butadiene-styrene triblock copolymers) (Cheng, 2002).

1.5 Composites

A composite material is a macroscopic combination of two or more different materials, having detectible interface between them. Composites are used in electrical, thermal, structural properties and environmental applications. Current composite materials are ordinarily optimized to get a particular balance of properties for a given range of applications. Therefore, composites normally have a fiber or particle that is stronger and stiffer than the continuous matrix phase. Different types of reinforcements as well have good thermal and electrical conductivity.

1.5.1 Composites Classification

Generally, composites are classified in two different levels:

- The first level is usually based on the matrix ingredient. The main composite is comprised of organic-matrix composites (OMCs), ceramic-matrix

composites (CMCs) and metal-matrix composites (MMCs). Normally, OMCs supposed to include two classes of composites: polymer-matrix composites (PMCs) and carbon-matrix composites (CCMC) (typically referred to as carbon-carbon composites). Commonly, CCMC are formed from PMCs by involving the extra steps of carbonizing and condensation the original polymer matrix.

- The second level is based on the reinforcement from particulate reinforcements, continuous fiber laminated composites, whisker reinforcements and woven composites as shown in Figure1.2 (Zweben, 1998). Generally, the substantial volume fraction of the reinforcement is about ~10% or more to provide a useful increase in properties. Thus, particulate reinforced composites include those reinforced by spheres, flakes, rods and many other forms of approximately equal axes. The whisker reinforcements, with an aspect ratio typically between 20 and 100, are often considered together with particulates in MMCs. Mutually, these are sorted as a discontinuous reinforcements, because the reinforcing phase is intermittent for the lower volume fractions typically used in MMCs. While continuous fiber reinforced composites include reinforcements having lengths much bigger than their cross sectional dimensions and a large amount of them include fibers that are similar in size (length) to a general dimensions of the composite part. Each layer of a continuous fiber composite usually has a specific fiber orientation direction as in Figure 1.3.

There is also filled structure. The filler particles are included for the purpose of cost decrease more willingly than reinforcement, these composites, generally, not considered as a particulate composite (Miracle and Donaldson, 2003; Zweben, 1998).

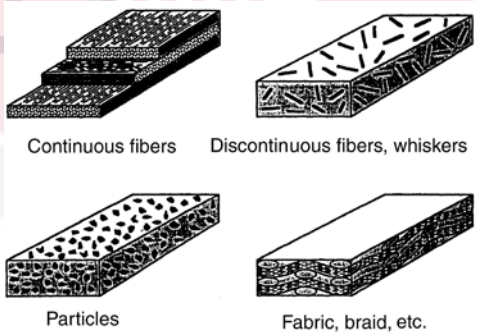

Figure 1.2: Common fiber reinforcement forms (Zweben, 1998).

1.6 Nanomaterials and Nanostructures

Nanomaterials (NM) are generally regarded as materials with a minimum of external dimension that measures less than 100 nm. or with internal structures measuring not as much of 100 nm. They possibly will be in the form of particles, tubes, rods or fibers. The nanomaterials that have the similar composition as known bulk form materials possibly will have different physic-chemical properties than the similar materials in bulk form, and may act in a different way if they enter the body.

1.6.1 Nanomaterials Classification

The production of materials and devices with new properties by method of controlling their microstructure on the atomic level has turned into a rising interdisciplinary field comprising of solid state physics, biology, chemistry and material science. The materials and devices included could be classified into the following three groups: The first group includes materials and devices with reduced dimensions in the shape of isolated, substrate-supported or embedded nanometer extent particles, thin wires or thin films. The second group includes materials and devices in which the nanometer extent microstructure is limited to a thin surface area of a bulk material, and the third category of bulk solids with a nanometer extent microstructure (Gleiter, 2000; Zweben, 1998).

One of the essential consequences of the materials science is the knowledge that most properties of solids rely on the microstructure. A decrease in the spatial dimension, or imprisonment of particles or semi-particles in a specific crystallographic direction inside the structure normally leads to changes in physical properties of the system in that direction. Therefore an additional classification of nanostructured materials and systems basically relies on the number of dimensions which exist within the nanometer range: zero dimensional structures, like nano-pores and nano-particles, one dimension, possessing only one dimension in the nanometer scale, and thus are as lamellae. Graphene and clays are known as being nano-layered particles. Two dimensions, the particles have two dimensions in the nanometer range and the third dimension is larger, their structure is extended. Cellulose nano-whiskers and carbon nanotubes are typical examples of this group. Three dimensions, when the particles have three nanoscale dimensions they are named ISO dimensional nanoparticles like spherical silica nanoparticles Figure 1.4 (Cheerarot, 2012; Pokropivny, 2007):

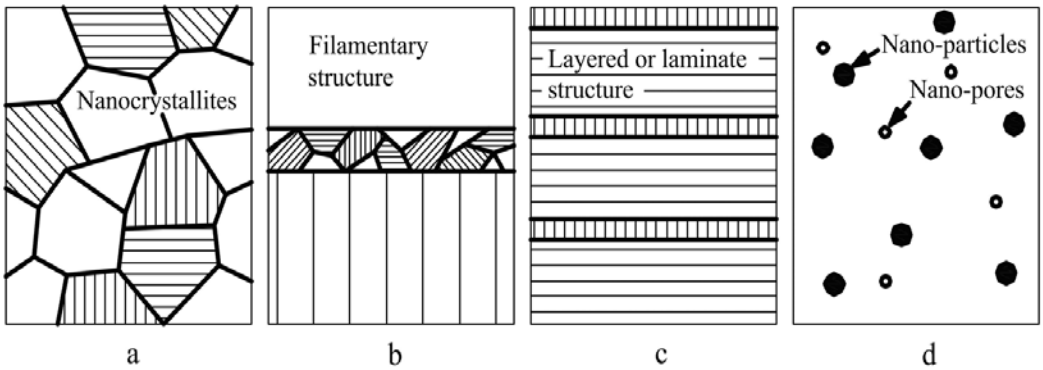

Figure 1.3: Schematic classification of nano – materials: (a) three – dimensional tructures;(b) two – dimensional; (c) one – dimensional; and (d) zero – dimensional structures (Pokropivny, 2007).

1.7 Polymer nanocomposites

Polymer nanocomposite (PNC) is a mixture of composites and nano-sized materials, it consists of two types of materials, a host polymer and nano-size filler. Polymer based nano composite is getting an increasing interest because of substantial enhancements in the physical and chemical properties of the materials. The logic behind these properties enhancement is the small size of fillers and homogeneous dispersion on the nano-scale within a polymer matrix in addition to the length scale of interaction with the polymer segments. There is a high volume fraction of an interface area between the polymer matrix and the nanoparticles because of the small particle size (Vaia and Giannelis, 2001). Therefore, a small amount of nano-filler can improve the polymer properties by several orders of magnitude. Also, the molecular mobility and packing density of the segments in that interface quite different from those in the matrix polymer (Kotsilkova et al., 2005). A few methods have been utilized to describe the structure and properties of PNCs like, X-ray diffraction (XRD), Scanning Electron Microscope (SEM), Fourier Transform Infrared Spectroscopy (FTIR) and transmission electron microscopy (TEM) (Chen et al., 2009; Olmos et al., 2014).

1.8 Polymer Degradation

The ultraviolet region in optical spectrum is the most harmful part of sunlight owing to its high energy. The photon energy related to it is of the order (from 0.3 to 0.4 MJ) , which is in the range of C—C energy, ultraviolet radiation could cause C—C bonds break, ensuing a scission of the polymer chain. Other bonds like C—O and C—H might also break. PMMA absorbs up to 95% of the incident UV rays within the range of UVC 100–280. The most important feature of arbitrary scission in a polymer chain is a fast decrease in the MW. The changes that happen in the physical properties of a polymer in addition to MW leads to decrease in its mechanical properties and its ability to perform satisfactorily in a few applications (Abouelezz and Waters 1978, Mitsuoka et al., 1993; Yousif et al. 2015). The arbitrary scission leads to the disintegration of the chemical bond to generate free-radical. There may be more than one free-root in a composition (Abouelezz and Waters, 1978; Rabek and Fouassier, 1989).

In PMMA, the number of scissions occurring in its chains relative to the number of quanta that absorbed by the chain and they recognized the proportionality constant as the quantum yield of chain scission (Abouelezz and Waters, 1978).

It was found that at room temperature random main chain fracture in PMMA degradation happens with ultra-violet UV radiation, while at higher temperatures the reaction is one of chain depolymerization. The energy amount that absorbed for each main chain fracture is about 550 eV with ultraviolet radiation, so that less than 1% of the quanta absorbed are effectual. In recent times, the wavelength sensitivity in photodegradation of PMMA was studied by monochromatic radiation using the Okazaki Large Spectrograph (OLS). It was discovered that the limit wavelength of main chain scission is between 260-320 nm.

Furthermore, the number of main-chain scission which has a greater value in the case of the irradiation with 280 nm. (Mitsuoka et al., 1993; Torikai and Hasegawa, 1998; Torikai et al., 1990).

1.9 Refractive Index

The refractive index (n) of the medium is a number describing how fast the light travels through a specific medium relative to light speed in vacuum. Generally, the refractive index defined by:

$$n = \frac{c}{v} \tag{1.1}$$

where c is the light speed in a vacuum, and v is the light speed in the medium.

The refractive index used as a measure of the material purity and optical design. In the field of transparent polymers, studying refractive index of the material gives a clear picture of the behavior of the penetrating rays in the material. The refractive index of the homogeneous material is isotropic even the molecules are oriented randomly (Kuzyk, 2006).

Practically, the refractive index measured directly by refractometer. Theoretically, it can find out from the optical band gap which obtained from the absorption curve edge of UV-VIS spectra. The absorption coefficient α can find from absorbance value $A = log\, {}^{I_\circ}/_{I}$ using the relation (Asha et al., 2009):

$$\alpha(h\nu) = {}^{2.303A}/_{T} \tag{1.2}$$

where T is the thickness of the sample, $I_\circ$ is the intensity of the incident beam and I is the intensity of the incident beam. The optical band gap determined from the curve tangent in the absorption edge for ($\alpha h\nu$) against ($h\nu$). The empirical relation between the optical band gap and refractive index proposed for different compounds by Reddy (Reddy at al., 1998):

$$n = \sqrt[2]{\frac{12.417}{Eg - 0.365}} \tag{1.3}$$

where Reddy equation is the modified form of Moss equation which proposed that the energy levels in a solid material are scaled down by the factor ${}^{1}/_{n^4}$. The Reddy equation compatible with practical results than Moss equation (Kumar and Singh, 2010).

1.10 Problem Statement

One of the major problems experienced in transparent polymers is degradation emerging from its exposure to sunlight (Ultraviolet radiation), which leads to changes in material color, surface glimmering and the cracks appearance of a surface. As a result, reduces the lifetime of the product. This issue up to some transparent polymers, such as PMMA and PSF, contain strongly absorbing chromophores as an integral part of their structure. The chromophores are capable to absorb UV ray energy and involve in the photochemical degradation reactions that occur via free radical mechanisms, leading to the formation of hydroperoxides and chain scission of the polymer. There are many researches which are based on blending the polymer as a solution of its problems. So choosing to blend PMMA and PSF with UV inhibitors in order to minimize photochemical interaction.

Unusual optical and thermal properties of eco-friendly cellulose esters motivate us to use them as an inhibitor for photochemical interaction in addition to the high-formation ability. Moreover, indium oxide nanoparticles (nano-In_2O_3) show a strong absorption below 450 nm with an absorbance peak at round 288 nm, it may contribute to curb the photochemical interaction with the polymer matrix (Maensiri et al., 2008a).

1.11 Objectives

The goal of the present thesis is to improve two important transparent polymers (Polymethylmethacrylate-PMMA and Polysulfone-PSF) in order to protect themselves and covered surfaces from the impact of ultraviolet radiation by using some environmentally friendly cellulose esters with nanomaterial. The objectives of this thesis:

1. To study the effect of adding different percentage concentrations of cellulose acetate derivatives (cellulose acetate butyrate CAB - cellulose acetate propionate CAP - cellulose acetate phthalate CAT) on absorbance and transmittance spectrum of PMMA and PSF blends separately in order to select less absorbency within ultraviolet region and high transmittance in the visible region, with taking into consideration the transparency of samples.

2. To analyze bi-composite samples PMMA/nano-In_2O_3 and PSF/nano-In_2O_3 using UV-VIS spectrum and select maximum absorbance. Then, formulate tri-composite samples which consist of selected PMMA/cellulose acetate derivatives and PSF/cellulose acetate derivatives individually with the addition of the selected concentrations of nano-In_2O_3.

3. To study the morphologe of all selected samples beside pure PMMA and pure PSF in addition to study the mechanical, thermal and thermo-mechanical properties of all selected samples in addition to pure PMMA and pure PSF.

1.12 Thesis Layout

The thesis consists of five chapters of chapter one provides an introduction of the concepts and terminology as stated in the thesis with the problem statement and aims. Chapter two gives a literature review. Chapter 3 describes the materials and a sample preparation method, moreover includes a detailed explanation of the devices that used in the project. Chapter four illustrates the results of measurements have been reached and conclusions. Finally, Chapter 5 comprises the summary and future studies.

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