CHAPTER I

INTRODUCTION

1.1 Polymer Composite

Composite materials are the combination of two or more materials (i.e., reinforcing element, filler, and composite matrix binder) differing in form or composition. Polymer composite consists of a polymer matrix and second material which is embedded in matrix. The polymer matrix may be a thermosetting polymer like epoxy, or thermoplastic like polypropylene (PP). The second material may be organic or inorganic material [1]. The common role of the second material is to act as reinforcement, enhanced mechanical performance but in some case it reduces the cost and has little, or no effect on performance.

In general, polymer composite materials have an improvement, or the increase of the properties of polymer getting better. The application of materials (polymers composite) can be used in traditionally. For example, composites between polymers and carbon nanotube (CNT) are wildly used because their strength, stiffness, and conductivity can be improved by the addition of CNT [2-4].

The conducting polymer composite such as poly(acrylonitrile-co-butadiene-co-styrene) (ABS)/carbon black composites making the application of ABS are widly used [5-6]. Generally, ABS is used as an engineering plastic because it is good chemical resistant, high impact strength, and high electrical resistance. However, the level of conductivity can be increased when electrically conducting additives such as carbon black (CB) are added to the ABS resin. Thus the application of ABS can be used for circuit packaging.

Polybenzoxazine is the thermosetting polymer. It appropriated for making composite because of their high performance properties such as thermal stability and good fire resistant [7-9]. Therefore, polybenzoxazine is applied in various fields such as electronic materials [10].

Moreover, the composite between polymer and organic materials (biomaterial) are wildly used such as hydroxyapatite [11-13]. Since hydroxyapatite is a mineral which its chemical structure similar to the mineral component of bones and hard tissues in mammals. It has been used for re-construction and repairing bone defects [14].

1.2 Polymer Composite Characterization

The study of polymer composite involved many aspects, such as the identification and characterization of the individual components presenting in the composite. It was well known that the mixing between the filler and the polymer matrix had influence on the mechanical properties of polymer composite [15]. The reinforcing (filler) efficiency in composite depends strongly on the uniform dispersion of reinforcment throughout the polymer matrix thus the characterization of homogeneity dispersion of composite material is important [16]. In gerneral, transmission electron microscopy (TEM) [1-3], atomic force microscopy (AFM) [5, 29], scanning electron microscope (SEM) [17-18], X-ray diffraction (XRD) [19-20], and tribological probe microscope [21], were used to characterize the filler and their dispersion in the matrix. These techniques give the information only about the distribution of elements but the changes in chemical bonding or chemical structure of material cannot be obtained [22-23]. Nevertheless, some techniques have limitation. For SEM analysis, the analyzed sample is coated with gold metal, thus the cost of analysis is high and sample is destructive. The TEM analysis, the specimen must very flat and thin (~0.1 μm), thus the sample preparation for studying polymer surface is difficult.

Fourier Transform Infrared Spectroscopy (FT-IR) and Raman spectroscopy were used to characterize the mixing of the composition of composite [22, 24-26, 27, 34, 37-38]. Raman spectroscopy is limiting use for polymer composite because many polymers give strong fluorescence. The characteristic scattering of the sample is obscured. Although the Fourier transform (FT) Raman spectroscopy has proved in this respect because the fluorescene is reduced, it was not completely eliminated. Fourier transform infrared (FT-IR) spectroscopy is one of the important techniques because this technique gives the chemical information and molecular structure of composition in polymer composite. The technique can be applied for studying of polymer composite in order to determine the chemical nature of different species.

1.3 Fourier Transform Infrared (FT-IR) Spectroscopy

FT-IR spectroscopy is the well-known technique for characterization of materials such as polymer composites. It is performed by irradiating a sample with an infrared beam. Some of the radiation is absorbed by the functional groups present in matter. Moreover, the intensity of absorbance is proportional to the concentration of the absorbing molecule. Thus FT-IR spectroscopy provides information both of the molecular species and their concentrations in a sample. The FT-IR spectroscopy can be measured in transmission and reflection modes (*i.e.*, specular reflection, diffuse reflection, attenuated total reflection, and reflection/absorption). The specular reflection technique is difficult for studying polymer surfaces. The reason is that the rough nature of the surface means reflectance spectra tend to be a mixture of specular and diffuse reflection, therefore there can appear distorted spectrum and it is difficult to interpret. The transmission measurements can be characterized if a section of polymer about 10 μm thick is cut from the sample. However, this technique is difficult and does not appropriate for polymer composite [26, 27]. These problems can be overcome by the use of an attenuated total reflection (ATR) Microspectroscopy [22].

1.3.1 Attenuated Total Reflection Fourier Transform Infrared (ATR FT-IR) Spectroscopy

ATR is a spectroscopic technique utilizing an internal reflection phenomenon uniquely established when light traveling in an internal reflection element (IRE) impinges on surface of a rarer medium with an incident angle greater than the critical angle. It is the technique for recording the optical spectrum of a material that contact with an IRE. Therefore, ATR spectroscopy is a surface-sensitive characterization technique. This technique has been employed to extract information, *e.g.*, functional groups, molecular orientations at the surface and interface regions of polymers. The technique possesses several advantages such as non-destructive sample, easy and fast operation, and having little or no sample preparation.

ATR spectrometry is suitable for the analysis various types of samples such as thin films deposited on a substrate, and polymer composites. ATR accessories employ an internal reflection element (IRE) of high refractive index (i.e., Ge, Si, ZnSe, or diamond). The difference of refractive indices between the IRE and sample is the causes of the infrared radiated to be totally internal reflected. At each reflection, the electric field penetrates into the sample by a short distance from the IRE/sample interface. Typically, the radiation penetrates the sample to a depth of only a few micrometers [27].

1.3.2 Limitations of ATR FT-IR Spectroscopy

For ATR FT-IR analysis, any sample that comes into contact with the IRE can be analyzed. Thus, the uniform sample-IRE contact is of a greater concern. The traditional ATR spectroscopy (macro methods), is desirable to achieve contact over the entire IRE surface. This requirement is easily achieved a good contact with liquid samples but obtaining good contact with solid samples will be difficult. Moreover, a large contact area (5 mm in diameter) resulting in an average of molecular information over a large sampling area was achieved. The changing of molecular information in small area cannot investigate.

1.3.3 ATR FT-IR Microspectroscopy

Infrared microspectroscopy is the union of microscopy and spectroscopy for microanalysis. Microscopy reveals a major role in selecting samples for analysis, observing the microstructure of sample and defining the microscopic area for analysis. Although this method has a limiting of spatial resolution with respect to other microanalysis method (*e.g.*, scanning electron microscope), the ability to provide molecular information and direct indentification has been the benefits of the method [1].

Infrared microspectroscopy has been employed for many years in primarily a qualitative capacity. However, recently developments in hardware and software have driven the method into the field of quantitative analysis. The imaging microscopes is straightforward to obtaining composition and information about the distribution of chemical species within the sample. However, the commercial IRE has limitation, the small IREs (μIRE) were developed. Ge and diamond were used as IREs. There are the slide-on Ge μIRE and the slide-on diamond μIRE. The μIREs have small contact area about 100 and 30 μm in diameter for Ge μIRE and diamond μIRE, respectively. Thus, the changing in small area of sample can be investigated.

FT-IR images can be obtained by mapping and the process is automated with a computer controlled motorising stage. The consecutive measurements on an array of points on the sample according to obtain a map of spectra which can be used to generate chemical images. FT-IR spectroscopic imaging has an advantage comparing to other imaging methods for the characterization of materials because it relies on the characteristic absorption corresponding to molecular information in the sample [22-23]. Other advantages of ATR FT-IR imaging are required minimal, or no sample preparation, due to the penetration depth of IR light in the sample is independent of sample thickness.

1.4 The Objective of The Research

The objective of this research is to develop technique for characterizing the homogeneity of polymer composites by ATR FT-IR microspectroscopy.

1.5 Scope of The Research

1. To investigate the homogeneity of polymer and polymer composite by using ATR FT-IR microspectroscopy. The experiment was divided into two procedures: (1) surface characterization and (2) depth profile characterization.

2. To determine the homogeneity of polymer and polymer composite by surface characterization with specular reflection technique.