1.0 INTRODUCTION

1.1 Background and Problem Statement

Fire protection is one of the most important fire safety aspects of building regulations. Fires within structures and buildings can result in catastrophic consequences. The normal problems caused by fire in a building include loss of structural integrity, rapid flame spread, high heat levels, smoke and toxic fume emissions.

A comprehensive fire protection program for the building involves the installation of active and passive fire protection systems. Active systems are aimed to actively combat fire at its onset, consisting mainly of extinguishers, water sprinklers, and fire detectors, among others. On the other hand, passive systems act as a form of second-line defense to contain the rapid spread of fire and ensure a particular prescribed fire resistance period is satisfied.

Passive fire protection of steel members is usually achieved by using materials such as cement-based sprays, board, batt materials and intumescent coatings (Wang et al., 2006). This research has focused on developing water-borne intumescent fire protective coating on steel. At ambient temperatures, intumescent fire protective coatings visibly look similar to ordinary paint finish. This fire protection relies for the most part on the unique ability of intumescent coatings to combat fire. Deterioration of the mechanical properties of steel in a fire can seriously compromise structural integrity, and cause rapid creeping, buckling, collapse or some other failure. Usage of intumescent coatings is highly recommended in building constructions as it can save precious human lives and assets. The critical temperature of steel is 550°C. Intumescent coatings are designed to perform under severe fire conditions and to maintain the steel's integrity between 1 and 3 hours (Duquesne et al., 2004; Jimenez et al., 2006a).

Following recent experimental work, several researchers have studied the mechanisms and characterization of the intumescent coatings properties (e.g. (Chou et al., 2010; Chuang et al., 2009; Dai et al., 2009; Maude et al., 2009; Wang et al., 2010). Intumescence is defined as the swelling of certain substances when they are heated or exposed to fire (Camino et al., 1989; Camino et al., 1990). The fire protection provided by intumescent coatings occurs by three reaction processes: (i) the coating material decomposes, (ii) inert gases evolved from the decomposition reaction are produced at a rate high enough to drive back hot convective air currents, and most importantly, and (iii) the coating expands into a highly porous char layer with a high resistance to heat conduction from the flame into the underlying steel substrate (Gu et al., 2007; Jimenez et al., 2006b; Toro et al., 2007).

Intumescent coatings contain 'active' ingredients bound together by a binder. Generally, three 'active' ingredients are used: an acid source (normally ammonium polyphosphate (APP)) or a mineral acid), a carbon source (such as pentaerythritol (PER) or polyols) and a blowing agent (normally melamine (MEL)). The formulation of the coating has to be optimized in terms of physical and chemical properties in order to form an effective protective char layer (Bourbigot et al., 2010). However, the char layer formed from APP-MEL-PER coating was easily oxidized and detached from the steel substrate at high temperature (Wang et al., 2006). Thereby, in order to maintain fire protection of the coating, it is necessary to improve anti-oxidation of the char layer.

The binder in the intumescent coating is important due to its two effects: it contributes to the formation of a uniform foam structure and char layer expansion (Wang and Yang, 2010a). The type of binders currently used in intumescent coating such as polyvinyl acetate emulsion, vinyl chloride latex, alkyd resin, epoxy resin and acrylic resin. These binders even have good performance in water resistance but their chemical compositions produce high amount of toxic gases and smoke when expose to fire. In this study, the water based epoxy emulsion and silica fume as mixed binder were used in order to minimize the smoke and toxic fume emission without compromising on quality and effectiveness of intumescent coating in fire protection.

Flame retardant fillers can strongly influence the combustion characteristics of the coatings, including its resistance to ignition, and the extent and nature of smoke and toxic gas emission products. This may result from simple dilution of the combustible fuel source, slowing down the diffusion rate of oxygen and flammable pyrolysis products and changing the melt rheology of the coating, thereby affecting its tendency to drip. Fillers undergo endothermic decomposition when they are exposed to heat, which cools the solid, or condensed phase, and release gases (H_2O and/or CO_2), which dilute and cool flammable combustion products in the vapour phase. The residue remaining after filler decomposition may also be highly significant in providing a thermally insulating barrier between the underlying substrate and external heat source, in addition to contributing to overall smoke suppression. Recently, there has been several attempts to use eggshell components for different applications, due to its chemical composition and availability, making it a potential source of environmentally-friendly filler for intumescent coatings (Toro et al., 2007). Chicken eggshell (CES) is an aviculture byproduct that has been listed worldwide as one of the worst environmental problems, especially in countries where the egg product industry is well-developed.

CES contains about 95% calcium carbonate ($CaCO_3$) in the form of calcite and 5% organic materials such as type X collagen, sulfated polysaccharides, and other proteins (Arias and Fernández, 2003).

Most buildings in Malaysia do not apply fire proof intumescent coatings because usage of such coatings escalates building costs. Current commercial intumescent coatings are very costly due to the materials used and will incur immense additional expenses in the building and construction line. Besides that, intumescent coatings are not as frequently used as active fire protection due to lack of knowledge and exposure to its potential as highly effective fire-resistive components. Existing intumescent coatings use alumina trihydrate (ATH) or magnesium hydroxide $Mg(OH)_2$ as a flame retardant filler and solvent based epoxy resin as a binder. Usage of solvent based epoxy resin produces high amount of toxic gases and smoke which results in negative impacts to environment. As yet, there is a lack of extensive research in the field of intumescent coatings and awareness of its role and ability in containment of fires.

This study aims to develop a novel intumescent coating that uses recycled by-products which enables significantly lowered production cost and yet has excellent fire-proof performance. Hence this intumescent coating is a low-cost and efficient solution for fire-safety regulations in buildings. Besides that, it has the potential to play a significant role in the conservation of ecosystems and minimization of environmental impacts.

1.2 Research Objectives

The purpose of this research is to develop a new intumescent fire protective coating by using recycled CES as a novel bio-filler, water-based epoxy resin and silica fume (SF) as mixed binders. SF is a low-cost industrial by-product with high thermal stability. Using an aviculture by-product (CES) and industrial by-product SF in the intumescent coating results in non hazardous and environmentally friendly invention that not only protects human lives but also improves the cost-effectiveness and fulfills the requirements of the high-performance fire protection.

The objectives of this research are listed as below:

1) To investigate the formulation and characterization of water-borne intumescent fire protective coatings.
2) To synthesize and optimize the fire protective efficiency of water-borne intumescent coatings for the protection of steel during fire tests.
3) To determine the effects of flame retardant components on the performance of intumescent fire protective coatings in terms of sticking ability, fire protection, thermal stability, bonding strength and surface morphology.

1.3 Scope of the Thesis

To achieve the research objectives, a series of experimental work were conducted. For this purpose, APP phase II was used as an acid source, MEL as a blowing agent, PER as a carbon source, SF and water-borne epoxy emulsion as a mixed binder and CES as a flame retardant filler. The water-based intumescent fire protective coatings studied in this work were prepared by using the solution blending method.

The coatings were characterized by thermogravimetry analysis (TGA), scanning electron microscope (SEM), field emission scanning electron microscope (FESEM), Instron microtester, Bunsen burner test and furnace test. TGA curves were used to determine the thermal stability and anti-oxidation of samples. From the TGA curves, the effect of filler and binder incorporation into the flame retardant additives can be examined. The results of TGA are useful to study thermal stability of the coatings and can reveal the loss of weight and decomposition of the materials under study. These parameters are important in understanding the variation of decomposition with the doping binder and filler.

The fire protection performances of the intumescent coatings prepared in this work were studied using the Bunsen burner and furnace tests. Bunsen burner is used to characterize the formation of char layer and sticking ability of the coatings during burning. Moreover, the furnace test was controlled to closely follow the Eurocode parametric time-temperature curve. This time temperature curve measurements are often primarily performed for the determination of the fire-resistance rating of a sample.

The surface morphologies of the coating and char layers were studied using the SEM and FESEM test. The results of SEM and FESEM are useful to evaluate porosity of the surface structures. In the second part of the study, the pull off test was used to determine the bonding strength of the coatings to the substrate.

1.4 Organization of the Thesis

This thesis consists of five chapters. Chapter one describes the background and problem statement, followed by an outline of the research objectives, the scope and organization of the thesis. Chapter two presents the research studies on relevant past flame retardant materials and intumescent fire protective coatings for the protection of steel during fire tests. The mechanisms of flame retardant additives or fillers at elevated temperatures are mentioned. A brief review of existing standard fire curves recommendation from Eurocode, International Organization for Standardization (ISO) 834 and American Institute of Steel Construction (AISC) specifications are presented. In addition the mechanical properties of steel at elevated temperature are also discussed.

Chapter three describes the materials properties and test program utilized for this research, including the physical and chemical properties of flame retardant components, sample preparation method and the characterization techniques of intumescent coatings.

Chapter four describes qualitative and quantitative determination of specimen characteristics during small scale lab tests. The physical and chemical mechanisms of the samples are also studied. The overall discussion on the results of the present research work is given in this chapter as well.

Finally, conclusion of the research and recommendations for future research is given in chapter five.