



Innovative precursor for manufacturing of superior enhancer of intumescence for paint: Thermal insulative coating for steel structures

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ABSTRACT

The represented study is a compendium on innovation of new precursor for enhancing the intumescence properties of paints, as thermal insulative coating for steel structures. Herein, intumescence is superiorly enhanced via the employment of phosphorous containing copolymer to act as fire-retardant additive for steel paint. The as-mentioned copolymer was synthesized via emulsion polymerization between phosphated 2-HEMA as a phosphated monomer and three other acrylic monomers. The methodologies for synthesis of phosphated 2-HEMA and its usage in emulsion polymerization with acrylic monomers for preparation of phosphorous containing copolymer, were illustrated. No reports had formerly studied the usage of phosphated 2-HEMA, as phosphorous containing monomer, in emulsion copolymerization, for preparing flame retardant additive of intumescent paint. Both of phosphated 2-HEMA and the so-produced phosphorous containing polymer were characterized via the interpretation of physical properties, FTIR, EXD and particle size distribution data. The synergistic effects for the concentration of phosphated 2-HEMA applied in preparation of the copolymer, as a flame-retardant additive were studied. Flame retardancy properties of the as-synthesized copolymer were performed by measurements of Limiting Oxygen Index, test of the direct flame, adhesion test (ASTM 3359) and vertical/horizontal fire spread (UL-94). The fire resistance of steel structures after coating with the intumescent paint, prepared by employment of the as-synthesized copolymer to be as a binder, was confirmed using scrub (ASTM D2486) and fire resistance (UL-1709) tests. All of the performed measurements approved the positive evidence that the as-synthesized phosphorous containing copolymer can be applicable as flame-retardant additive; superior enhancer of intumescence properties of paint.

1. Introduction

Thermal protective materials are employed for slowing down the combustion processing, heat transferring to the structural elements, and delaying the effect of temperature variation on its resistance. Fire resisting materials can be employed as a physical barrier, in order to reduce the energy transferring rate such as masonry. Where, heat transferring is proceeded physically through the conduction (statically) and the convection (moving means). If it does not occur physically, it could be taken a place by radiation, as the energy could be transferred by electromagnetic waves. Another possibility for fire protection is through endothermic chemical interaction. As by exposure to high temperatures, the as-chosen chemical reactants will undergo endothermic reactions, in order to absorb a great portion of the thermal

energy, to produce a new thermal protection interface. An example for the so-mentioned chemical reagents is the plaster, which as it acts as a fire-resistant material through controlling the chemical release of water, to be in turn acts as a thermal barrier. The as-produced layer will act in protecting the coated structure to remain at a constant temperature around 100 °C [1].

Using of steel structures in constructions has several advantages, however, the building blocks which contains such materials also have specific shortcomings, as firing of steel is subjected to deflection with exposing to high temperatures [2]. It has been reported that, using of steel structures without fire protective coatings disintegrate when exposed to heat up to 450–550 °C [3]. For solving this problem, steel structures are coated with different heat resistant coatings for provide heat-resistance property against 1100 °C temperature for the duration

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of 1–3 h [4]. Fire protective coatings used for steel structures are grouped into two basic categories: passive (plaster, rock wool, gypsum) and active (fire protective or intumescent paint) coatings [5]. Fire resistant substrates used to protect steel structures functionalized through acting as a fire barrier in order to prevent fire from spreading [6].

Intumescent paints are examples of thermal resistant materials as it is ascribed to employ through as chemical interactions. As it has been subjected to elevated temperatures, it starts to undergo various endothermic reactions that results in the formation of a charred foam with high insulation features. The advantages of the thermal protection through chemical interactions using intumescent paints are: i) it mainly applied as traditional decorative paint with different colors, good surface finishing and durability; ii) the application of such type of paints is simple in addition to easy maintenance properties and also it does not require any additional substrates for attaching to the structure; iii) it can also be applied in the protection of structural connecting areas; in addition to; iv) it does not effect on the intrinsic features of the as-coated surface (as mechanical properties). Therefore, it can be uploaded on to building structures without loss or modification of structural capacity [1].

According to Troitzsch [7], intumescence is proceeded by the function of the following components: 1) *acid Source*: which is mostly a salt of a non-volatile inorganic acid, such as boric, sulfuric or phosphoric acid, which release their acid at temperatures above 150 °C, and the as-generated acid acts in initiating series of chemical reactions, which is beginning with dehydration of carbonaceous compounds and their charring; 2) *carbonaceous compound*: a compound with an numerous of hydroxyl radicals, which is supposed to be dehydrated when exposed to etching via a reaction of esterification, such like; pentaerythritol, amide, and urea-formaldehyde or phenolic resins; 3) *foaming compounds*: under the influence of elevated temperatures, such compounds act in releasing large quantities of non-flammable gases such as hydrogen chloride (HCl), ammonia gas (NH₃) and carbon dioxide (CO₂), to form a foam with an aspect of carbonized material on the substrate. For this purpose, compounds such as chlorinated paraffin, melamine or guanidine are used, then; 4) *binding resins*: such substrates are responsible for preventing the dispersal of such evolved gases. Chlorine-based rubber is a one of highly recommended materials to be applicable in such purpose.

Melhado [8], reported the basic concepts for illustrating the behavior of steel structures by exposing to elevated temperatures and presented a new approach for protection criteria. Silva [9] also studied the behavior of steel structures by exposure to high temperatures. Additionally, Abreu and Fakury [10] developed a new program for determining the temperature of structural elements with and without protection. Martins [11], also represented a report for estimating the critical temperature of bars exposed to normal compression forces and beams. However, Lopes Ribeiro [12], had considered with studying the accuracy of regulatory procedures for controllable distribution of thermal energy in the cross section of structural elements under elevated temperatures.

In recent years, preparation and characterization of new products for improving the flame retardancy have attracted the attention of academic researchers interested in such field [13–19]. Among the most reported flame retardant systems in literature are nano-clays [20,21] and phosphorous compounds [22]. Particularly, the latter class of flame retardants comprises either inorganic or organic structures [23], also it can act in the vapor phase or in the condensed phase, also sometimes can be functionalized simultaneously in both phases [24].

Acrylics are difficult to be hydrolyzed, well recognized to exhibit good weather resistance [25] and are commonly utilized for coating applications due to their outstanding outdoor durability. Additionally, it is well known for its excellent properties such as toughness, flexibility, resistance to chemical fumes, acids, alkalis and water as well as gloss retention [26]. Therefore, these polymers are primarily made in emulsion or solution, recent reports were considered with synthesis of

Table 1
Chemical composition of blank copolymer and the so-prepared phosphorous containing copolymers.

Sample	Composition
Blank copolymer	A copolymer of Methacrylic acid, methyl methacrylate, butyl acrylate and hydroxy ethyl methacrylate.
T1 Copolymer	A copolymer of methacrylic acid, methyl methacrylate, butyl acrylate and 2.5% of phosphated 2-HEMA
T2 Copolymer	A copolymer of methacrylic acid, methyl methacrylate, butyl acrylate and 5% of phosphated 2-HEMA
T3 Copolymer	A copolymer of methacrylic acid, methyl methacrylate, butyl acrylate and 10% of phosphated 2-HEMA

Table 2
Characterization and physical properties of blank and the so-produced phosphorous containing copolymer.

Copolymer	Color	Viscosity (cp)	Density (kg/L)	Solid content (%)	pH	Minimum film forming temperature (MFFT, °C)
Blank	White	2000	1.06	49.7	6.3	3.7
T1	Yellowish	2500	1.06	49.8	7.0	0.0
T2	Yellowish	17000	1.06	49.8	7.2	–1.5
T3	Yellowish	20000	1.06	49.9	7.5	–2.5

acrylic polymers to be applicable in different purposes [27–29]. In acrylic monomers containing phosphorus are great potential candidate for the flame retardant modification of steel structures due to the key role of P synergism in flame retardancy. Therefore, the objective of this study is to characterize the efficacy of flame retardation obtained by water borne (emulsion) intumescent paint as a thermal insulative coating for steel structures.

The current study represents a new approach for manufacturing superior thermal insulative/intumescent steel paint by exploiting phosphorous containing copolymer as a fire-retardant additive. The so-mentioned copolymer was prepared via emulsion polymerization of a mixture from phosphated 2-HEMA as phosphorous containing monomer and three other acrylic monomers. According to literatures, no reports had studied the usage of the phosphated 2-HEMA, as phosphorous containing monomer, in emulsion copolymerization, for preparation of flame retardant additive of intumescent paints.

The chemical formula of the phosphated 2-HEMA was confirmed by FTIR spectra. However, the solid content, viscosity, pH, color changes, density, minimum film forming temperature, particle size distribution and energy dispersive X-ray (EDX) analysis were all detected and clarified for the as-produced phosphorous containing copolymers. The fire retardancy of the as-synthesized copolymer was assessed by estimating the limiting oxygen index (LOI). Furthermore, studying the effect of fire on the primer, the adhesion test and vertical/horizontal fire spread tests have been all exploited for evaluating the flame retardant features of the as-prepared copolymer. Lastly, the capability of the so-produced copolymer as additive as a superior enhancer of intumescence, to be applicable as a thermal insulative coating for steel structure was approved through scrub test and fire resistance test for the so-coated steel materials.

2. Materials and methods

2.1. Materials

2-hydroxy-ethyl methacrylate (2-HEMA) (Mwt = 130.1 g/mol; Evonik industries; Germany), Phosphorus pentoxide (P₂O₅), Mono-methyl ether hydroquinone (MEHQ) (inhibitor; BASF SE petrochemicals; Germany), Sodium bicarbonate, sodium dodecyl benzene sulfonate (surfactant; 98% active flakes; Rahodia; North America), and

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