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SYNTHESIS AND CHARACTERIZATION OF ECO-FRIENDLY TEXTILE COATINGS USING ZNO NANOPARTICLES, POLYVINYL ALCOHOL, AND AMMONIUM POLYPHOSPHATE

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Abstract:
Textile plays an important role in the economy of this world. Goods such as functional and intelligent textiles, in particular, garments with built-in multi-functional features such as flame resistance are needed to improve living conditions. ICSS 2018 Textile materials used in everyday life, hospitals and factories represent potential fire hazards due to the ignition of textile fabrics or combustion. As a result, textile manufacturers are focusing on the development of application specific textile finishes and coating to desserts for needs of consumers globally. Various natural and artificial textile fibre which includes cotton, polyester, woollon, jute etc. are susceptible to catching hearthplace and burning This work describes the development of flame-retardant textiles. In this study, zinc oxide nanoparticles (ZnO NPs) at 2 and 4 (wt%) were incorporated with polyvinyl alcohol (PVA), and ammonium phosphate (APP) was applied flame retardant. The textile coating was applied via the Dip-Pad technique. Flame retardancy assessment data suggest that when flame retardant agents are introduced, the burning rate of the coated textile decreases and that the effect is dose-dependent with respect to ZnO NPs contents. There is no sign of cracks on the surface of the textile as observed in scanning electron microscope imaging, and an adhesion between coating and fibers with a clear interfacial layer.

Keywords: Textile, Coating, ZnO Nanoparticles, Polyvinyl Alcohol, Ammonium Polyphosphate.

INTRODUCTION

Textiles use themselves in our daily life as we have to wear clothes and its a huge cost-effective product both because of its high adaptability and cheaper price due it color range. Based on the use of fibers, whether natural or synthetic alone or in combination with one another, textile fibers are therefore divided into four major groups: natural fibers (NFs) like those obtained from cotton, flax, silk, wool; regenerated fiber like viscose; synthetic fiber such as nylon, vinyl, polyester and acrylic; and inorganic fibers (glass or asbestos). However, the properties of these textiles need to be suited for their end use therefore it is essential to improve some textile properties such as UV resistance, antibacterial, flame retardant and thermal stability as well electrical and mechanical properties. Textiles are polymer-based and thus have abundant carbon in their chemical structure, rendering them highly combustible. Fabrics and upholstered furniture are, therefore, the first materials to ignite when ignited by sources like cigarettes and candles. Indeed, all these efforts are directed toward improving their properties such as flame-retardancy [1].
Polymer combustion was a complex process that involves multiple steps in the solid or gas phase and in the transition zone between them, a key transition phase in polymer combustion comes into play when a flame source or heat source increases temperature, which causes bond breakup and formation of volatile products, these then diffuse into ambient air creating an ignitable mixture fire (flammable) at those points where temperature rises above ignition point level. Combustion cycles related to polymers are depicted in figure 1 [2].

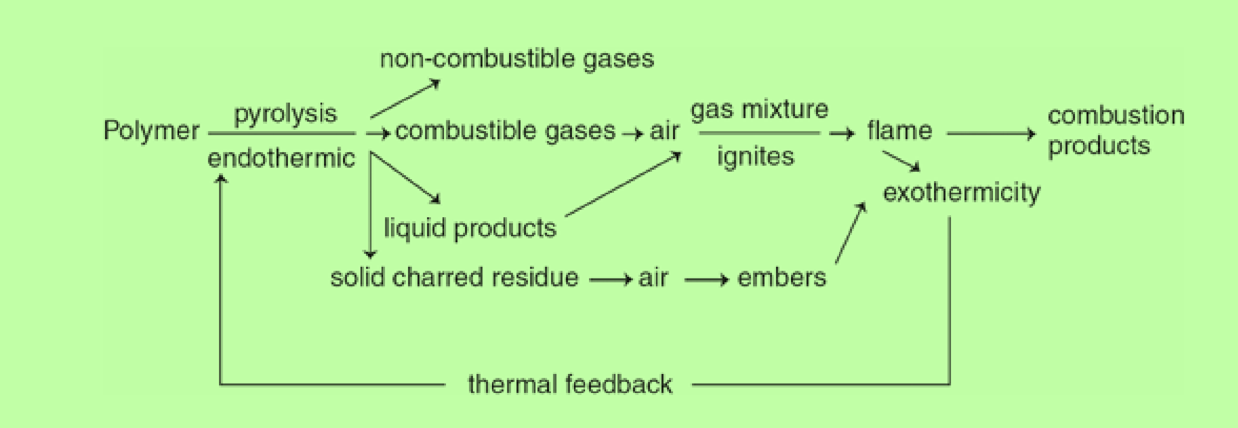

Figure 1. combustion cycles of polymers [3].

As previously stated, the combustion of polymers is closely associated with three key elements: combustibles, combustive agents, and heat, to effectively enhance flame retardance in polymers, it is crucial to disrupt one or more of the connections among these three elements, this disruption can be achieved through various methods, such as implementing an endothermic reaction that cools the polymers by absorbing heat or by emitting non-flammable gases to dilute the flammable gases, thereby reducing combustibles, another method involves encouraging the formation of char on the burning surface of the polymers, which effectively obstructs the interaction of the volatile products of polymer decomposition with the surrounding air. Generally, flame retardancy can function within the condensed phase or in the gaseous phase, thereby impacting the multiple processes involved in polymer combustion, including heating, decomposition, ignition, and the spread of flames [3].

A flame retardant (FR) is a substance, either inorganic or organic, that is added to flammable materials to enhance their resistance against catching fire, flame retardants function by stopping or, at the very least, slowing down the ignition of these materials, thus contributing to the preservation of lives and the safeguarding of property, these chemicals are employed to minimize fire hazards by decreasing the likelihood of materials igniting [4].

Flame retardant that is incorporated into polymer should:

1. Decrease the flammability of the polymer when contrasted with the unaltered polymer.
2. Diminish the amount of smoke produced under defined testing conditions.
3. Not elevate the toxicity of combustion products arising from the modified polymer in comparison to the unmodified polymer.
4. Exhibit an acceptable or minimal impact on other performance characteristics of the product [5].

Flame retardant systems are designed to prevent or halt the process of polymer combustion, the primary mechanisms through which these flame retardant systems operate are documented, in the Physical Mode, certain flame retardant additives undergo endothermic decomposition leading to a reduction in temperature due to heat absorption, this process cools the reaction environment thereby lowering the combustion temperature of the polymer, among the materials in this category are hydrated alumina and magnesium hydroxide, which release water vapor at around 200°C and 300°C, respectively. This significant endothermic reaction is recognized for its function as a "heat sink [3].

Flame retardancy can be achieved through the chemical alteration of the combustion process in both the gaseous and condensed states, the combustion process, which operates via a free-radical mechanism, can be interrupted by the addition of flame retardant substances that systematically release certain radicals (such as Cl• and Br•) into the gas phase, flames consist of a complex series of oxidation reactions, where the step that controls the rate is a branching event, here, molecular oxygen (O2) from the atmosphere interacts with a hydrogen atom (H•) released from the fuel to produce an oxygen atom (O), which is a highly reactive entity, and a hydroxyl radical (OH) . In the condensed phase, two distinct types of chemical reactions prompted by flame retardants can occur, Firstly, the flame retardants may facilitate the breaking of polymer chains. In such scenarios, the polymer melts and consequently moves away from the heat source. Alternatively, the flame retardant may induce the development of a carbonized, potentially expanded, or glass-like layer on the polymer's surface due to the chemical transformation of the degrading polymer chains. This charred or vitrified layer serves as a physical barrier that insulates between the gaseous phase and the condensed phase [3].

Numerous metal oxides exhibit multifunctional characteristics such as self-cleaning, antimicrobial properties, UV protection, flame resistance, hydrophobicity, and electrical conductivity, which are applicable in smart and functional textile , the flame resistance of zinc oxide is attributed to the condensed phase mechanism, which operates through the thermal barrier effect, Zinc oxide, known for its thermal stability, can form an insulating barrier on the textile surface, thereby impeding the combustion process by limiting the transfer of oxidants and fuels. Zinc oxide nanoparticles are encapsulated within a polymeric shell and can be applied to cotton, jute, and sisal fibers utilizing a dip coating method or a layer-by-layer dip coating technique to create textiles that are flame-retardant and UV protective. Moreover, the thermal stability of the treated fabric is significantly superior when compared to that of untreated fabric [6,7].

Metal hydroxides serve as efficient agents for suppressing smoke and retarding flames through decomposition processes, when textiles are coated with metal hydroxides, graphitization occurs, leading to the generation of incombustible gases that mitigate heat damage, in addition to metal oxides and metal hydroxides, layered double hydroxides (LDH) represent an innovative category of flame retardants for coatings used on polymers and textiles. LDH operates under a condensed phase mechanism by creating a heat-resistant barrier and facilitating the char formation process. [8,9]. Determine the combustion rate (v) in millimeters per second, using the equation provided [10].

$$V = 60\ L / t \qquad (1)$$

where:
L = the burned length, in millimeters.
t = the time, in seconds.

1. MATERIALS AND METHOD

Cotton/Polyester blend fabric 65/35 (152 g/m^2) were kindly supplied by Misr Company. Powdered polyvinyl alcohol (PVA) was sourced from Yonghui Chemical Holdings Limited, located in China.
Zinc oxide Nanoparticles (ZnO NPs) were supplied as a powder Alchemy Scientific with the properties is shown in table 1.

Table 1. The Specification of Zinc Oxide Nanoparticles (ZnO NPs).

Properties	Value
Color	White
Particle Size	30-50 nm
ZnO content	>> 99.6%

Ammonium phosphate APP was supplied by Shian Chem company with properties as shown in table 2.

Table 2. The Specification of Ammonium phosphate APP.

Properties	value
Chemical formula	$(NH_4PO_3)_n$
color	white
Nitrogen proportion (N)	14–15%

2. EXPERIMENTAL PART

Firstly: we perform a preliminary treatment on the fabric composed of cotton and polyester, as it is a fairly inert substance, therefore, it is necessary to enhance the adhesion by cleansing it with a solution of ethanol and water to eliminate impurities. Following this, we conduct a gentle alkaline treatment using a sodium hydroxide solution (NaOH) at a temperature of 60 C° for a duration of 20 minutes.
Secondly: PVA solution were prepared by dissolving 7 wt% of polyvinyl alcohol (PVA) in distilled water at a temperature range of 87-90°C. Continuous stirring is performed until the solution becomes clear, utilizing a magnetic stirrer, after which it is allowed to cool down to 55°C. Subsequently, zinc oxide at concentration (2, 4 wt%) is integrated into the PVA solution by incorporating 3 wt% of ZnO and employing an ultrasonic device to ensure thorough dispersion for a duration of 25-30 minutes. To mitigate clumping and to enhance flame resistance 12 wt% of ammonium polyphosphate (APP) is introduced, rendering the system an effective flame retardant.
Thirdly: The application of paint to the fabric is conducted through the Dip-Pad technique, which involves submerging the fabric in the flame-retardant mixture for a duration of 10 minutes, afterwards, the fabric is squeezed out to achieve a wet pickup percentage ranging from 75% to 80%. The next step involves drying the fabric at a temperature of 100°C for 10 minutes, followed by a heat-curing process at 140°C for 5 minutes as shown in Figure 2.

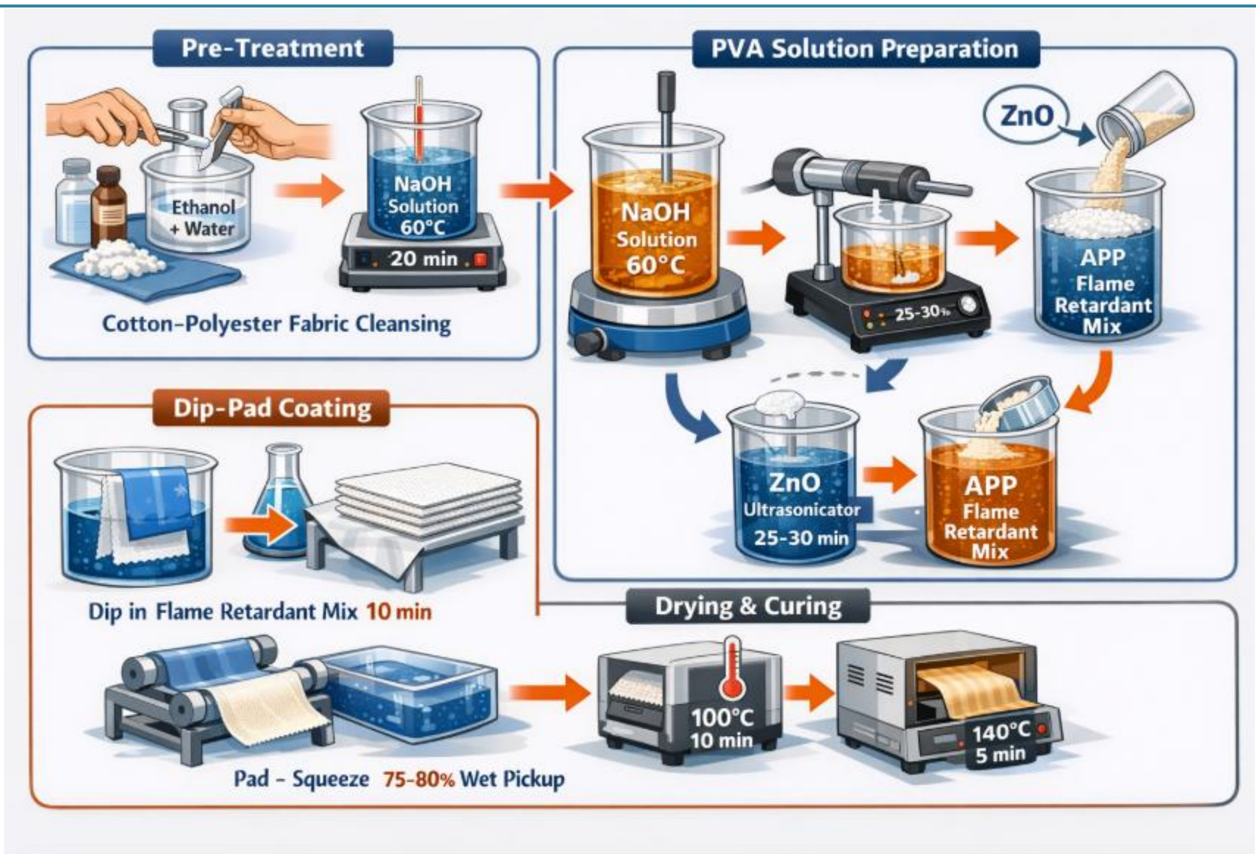

Figure 2. Laboratory preparation of flame-retardant textile.

3. RESULTS AND DISSECTION

3.1. Flame retardancy test

The flame-resistant properties of the Cotton/Polyester textile coatings that were developed were evaluated using horizontal (ASTM 635-03) testing method, the specimens were cut to a size of 125 mm in length and 13 mm in width. Each specimen was marked with two lines that are perpendicular to the lengthwise axis, at positions of 25 mm and 100 mm. The specimen was secured at the end that is farthest from the 25 mm reference line. Then a flame was applied at an angle of 45 degrees for a duration of 30 seconds without altering its position; afterward, the flame was removed, and the elapsed time (t) was recorded in seconds, along with the measurement of the burned length in millimeters. The burning rate (v) was calculated in mm per second. Table 3 presents the findings of the combustion rate (v) acquired from the flame test.

Table 3. **Burning rate for textile coating.**

Specimens	L	t (Sec)	V (mm/Sec)
Textile	11	31	21.29
Textile -2wt% ZnO NPs $^+$PVA$^+$APP	7	32	13.125
Textile - 4wt% ZnO NPs $^+$PVA$^+$APP	5	35	8.57

The preceding table illustrates that textiles without the incorporation of flame retardant agents are highly susceptible to ignition in the presence of air. However, the inclusion of ZnO nanoparticles, PVA, and APP as flame retardant agents effectively inhibits the combustion of textile coatings. A minimal amount of ash was observed post-extinguishment, specifically at a concentration of 2 wt% of ZnO nanoparticles, when the concentration of ZnO nanoparticles was increased to 4 wt%, a noticeable reduction in the burning rate of the textile coating composite was evident as in figure 3, attributed to a substantial release of CO_2 and H_2O that facilitates self-extinguishment of the fire. This attributed to the fire suppression system comprised of Zinc Oxide Nanoparticles (ZnO NPs), Ammonium Phosphate (APP), and Polyvinyl Alcohol (PVA) operates through a synergistic interaction among these three components, where each plays a distinct yet supportive role: Function of Zinc Oxide Nanoparticles: These nanoparticles serve as a catalyst and a thermal stabilizer, they hasten the development of a charcoal layer, thereby enhancing its efficacy as a thermal barrier. Additionally, nanoparticles help to fill the spaces within the charcoal bed, minimizing both heat and gas flow. Radical Scavenging Inhibition: During the combustion process, free radicals such as H_2O and OH_2O are generated, which serve to impede the chain reaction of combustion. additionally, Ammonium Phosphate (APP) is recognized as the crucial element for flame retardancy, upon exposure to heat, APP undergoes decomposition, releasing Phosphoric acid (H_3PO_4) and non-flammable gases like NH_3. The phosphoric acid acts to dehydrate cellulose in cotton, resulting in the formation of a char layer that effectively insulates against heat. This layer also restricts oxygen from reaching the flame and decreases the emission of flammable gases, and the polyvinyl alcohol (PVA) operates as a binding agent and a source of carbon. It facilitates the adhesion of materials to the fabric's surface upon heating, it decomposes and aids in establishing a carbon structure, which fosters the creation of a coherent layer with adhesive properties.

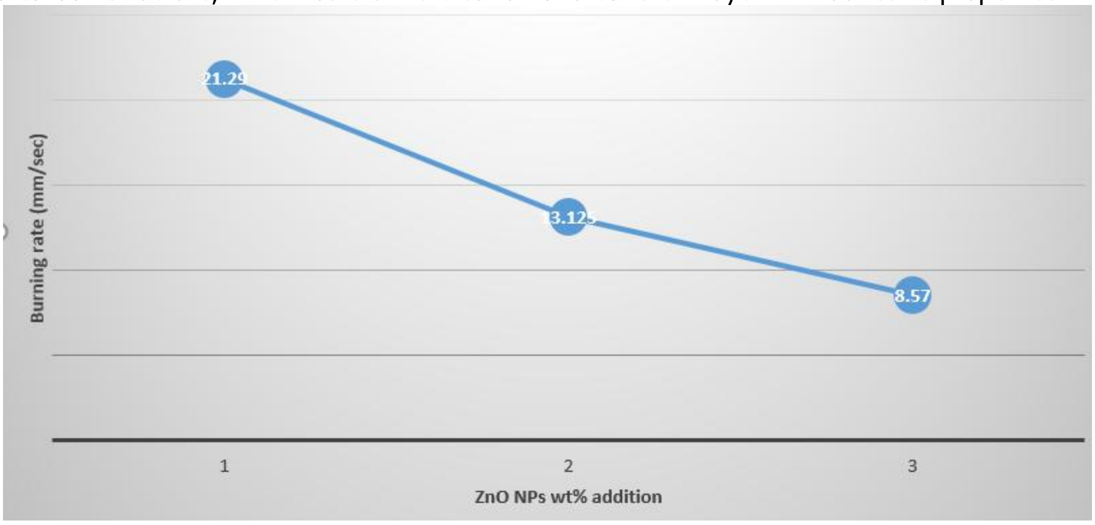

Figure 3. Burning rate for textile coating.

3.2. SCANNED ELECTRON MICROSCOPY (SEM)

A scanning electron microscope (JSM 840, Jeol) was utilized to examine the surface morphology and the cross-section of the coated textile. Prior to the observation, the samples were coated with a gold film using an ion coater E 5000 from Polaron Equipment Co, figure 4. It was noted that there was no development of cracks on the surface of the textile. Additionally, a robust adhesion between the coating and the fibers was identified, there were no notable cracks or delamination present, the coating fits closely to the contours of the fibers and demonstrates effective bonding through hydrogen bonding (PVA) and physical anchoring techniques.

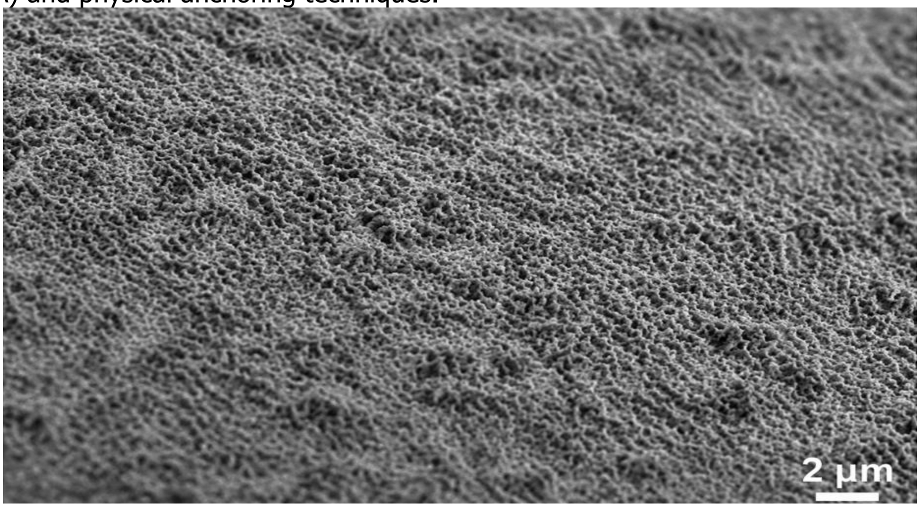

Figure 4. Scanned electron microscopy of textile coating with flame retardant agents.

4. CONCLUSION

Contemporary textile sectors emphasize the use of unconventional, structurally intelligent, and efficient materials for the production of multifunctional woven and non-woven textiles and fiber substrates. From this study, the following findings were drawn:

1. The incorporation of Zinc Oxide nanoparticles, Polyvinyl Alcohol, and Ammonium Polyphosphate will reduce the combustion rate of Cotton/Polyester fabric.
2. Polyvinyl Alcohol functions as a flame-retardant substance by releasing a substantial amount of water and carbon dioxide, which aids in extinguishing flames.
3. Scanning Electron Microscopy shows no signs of cracking on the textile surface, and a distinctly robust adhesion is observed between the coating and the fibers.

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