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Fire retarding properties of nanocomposites from polystyrene waste and antimony, barium, and nickel oxides' nanoparticles

Sabo Yasser Takko*, Boryo Doris Ezekiel Amin, Chindo Istifanus Y and Habib Abubakar Idris

Department of Chemistry, Abubakar Tafawa Balewa University, Bauchi, Nigeria

ABSTRACT

This work involves the synthesis of oxide nanoparticles via the chemical precipitation method and formation of nanocomposites by the *in situ* polymerization method. The treated and blank samples were prepared and investigated for ignitability, flame propagation, and afterglow time. Sb_2O_3/polystyrene (PS) was found to give a good effect for ignitability after 6.00 seconds than BaO/PS and NiO/PS nanocomposites and untreated PS after 5.00, 4.00, and 2.00 seconds as the concentration of the nanoparticles incorporated in the PS increased, respectively. Sb_2O_3/PS gave a better effect for flame propagation than untreated PS, BaO/PS, and NiO/PS nanocomposites. The Sb_2O_3/PS nanocomposite has an afterglow time of 1 second at 0.25 g concentration than BaO/PS (2 seconds) and NiO/PS nanocomposite (2 seconds) and blank PS (5 seconds). Conclusively, with this result, Sb_2O_3/PS nanocomposite was found to give a better retarding effect. It is recommended that during the production of polymer nanocomposites, PS/Sb_2O_3, PS/BaO, and PS/NiO nanocomposites could be used to hinder or slow down the propagation of fire during accidental fire outbreaks.

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1. Introduction

Polystyrene (PS) is one of the major synthetic thermoplastic polymers in use today. It represents one of the oldest and the most widespread polymers in the world. It started as far back as 1839 when a German apothecary Edmon Simon distilled an oily liquid named styrol from the resin of Turkish sweet gum trees. After several days, the sterol converted into a jelly product that he thought resulted from the oxidation process. For that reason, the jelly product received the name styroloxide (Cole, 2014). PS is a synthetic aromatic hydrocarbon polymer made from the monomer styrene (John and Duane, 2003). General purpose PS is clear, hard, and rather brittle. It is an inexpensive resin per unit weight (John and Duane, 2003). It does not have a sharp melting point because it is amorphous (Cole, 2014). This is observed in the gradual softening of the material over a wide range of temperatures. *T*g of the PS is between 74°C and 105°C, and below its *T*g, PS is hard and brittle (Cole, 2014). The disposal of PS wastes creates environmental contamination due to their non-biodegradable nature (Maharana et al., 2007). In the world today, thousands of PSs are being disposed of as solid wastes into the environment having paralyzing effects on the lives of people and the amount is progressively increasing every day/year (Cole, 2014). The increasing growth in the amount of PS wastes into the environment is due to the booming development of electronic products. The disposal of PS wastes creates environmental pollution and contamination due to their non-degradable nature. Scientific

* **Corresponding author** Y. T. Sabo Abubakar ✉sabotakkoy@gmail.com ✉ Department of Chemistry, Abubakar Tafawa Balewa University, Bauchi, Nigeria.

research had shown that the solution to these problems is achieved through the recycling method and transforming them to smart and useful materials. This research work desires to provide information on transforming PS wastes into nanoproducts with fire retardation properties for possible application in electronic appliances, buildings, and automobiles.

Apart from the numerous and multiple advantages and usefulness of PS in buildings, electronics, and other applications, situations and circumstances where PS wastes are recycled and transformed into useful materials are rarely found. Transforming PS wastes into useful materials reduces the effects of environmental pollutants on animals, plants, and humans. It also prevents wastes from entering into landfills and sites containing wastes. The negligence of transforming PS wastes into useful materials has attracted a lot of attention in the field of research, whereas the recycling of polymers to nanoproducts with fire retardation properties has not been adequately and sufficiently studied. Therefore, this research work will bring about knowledge that is of utmost importance in the field of research.

The aim of this research is to convert PS wastes to nanoproducts with fire retarding property. The objective of this research is to produce PS nanocomposites with fire retarding property for possible application in buildings, electronic appliances, and automobiles.

PS is also quite often used as a polymer matrix for the preparation of polymer-based nanocomposites. Polymer nanocomposites are hybrid organic–inorganic materials with at least one dimension of the filler phase less than 100 nm. Nanoparticles were considered to have a size range between 1 and 100 nm (Mittal, 2010). However, it has been shown that particles larger than 50 nm show no significant important of a materials properties as expected (Mittal, 2010). Novakov et al. (2013) suggested that the main task during the preparation of polymer nanocomposites is the production of aggregation of the nanoparticles and formation of larger size agglomerates or undesirable assemblies. These occur due to the higher interfacial reactivity of nanoparticles and their high surface tension energy (Novakov et al., 2013). Technological contributions in the areas of gas barriers, reinforcement, and flame retardancy have also been extensively exploited (Zammarano et al., 2005). For example, heat-resistant polymer nanocomposites are used to make fire fighter protective clothing and lightweight components suitable to work in situations of high temperature and stress. These include hoods of automobiles and skins of jet aircrafts, as opposed to heavier and costlier metal alloys. They can also replace corrosion-prone metals in the building of bridges and other large structures with potentially lighter and stronger capabilities (Bourbigot et al., 2002; Schubel et al., 2006).

Nanocomposites can be prepared by *in situ* synthesis of inorganic particles or by dispersion of fillers in a polymeric matrix (Khodaei et al., 2011). The synthesis of polymer nanocomposites usually applies bottom-up or top-down methodologies. In the bottom-up approach, precursors are used to construct and grow, from the nanometric level, well-organized structures. The block assembly or building block approaches can also be used, where already formed entities or nano-objects are hierarchically combined to originate the desirable material. The building block approach has an advantage compared to *in situ* nanoparticles formation, since at least one structural unit is well defined and usually does not have significant structural changes during the matrix formation. Chemical processes, such as sol-gel, chemical vapor deposition, template synthesis, or spray pyrolysis, are employed as bottom-up methodologies (Cai et al., 2006; Khodaei et al., 2011).

Polymers are used as flame retardants to enhance the ignition resistance of materials and to decrease the spreading of flames by ensuring minimum degradation of the polymer's properties. Flame-retardant materials can be classified into the following three categories: halogenated flame retardants, halogen-free flame retardants, and nanoadditives (Metehen and Mustafov, 2017).

The flame retardant is mainly used to slow down the burning rate rather than producing a non-combustible resultant product. It is used to decrease the fire danger caused by the use of polymers for particular applications. A small fire which could cause a major calamity can be prevented with the use of flame retardants (Metehen and Mustafov, 2017).

Flammable gases mix with air and ignition occurs. This causes the combustion process of polymers comprising the endothermic pyrolysis. Heat release is observed with exothermic process of flame propagation. In the condensed or vapor phase, flame-retardant additives can become active physically and/or chemically (Tkar, 1981). During heating, pyrolysis, ignition, or flame spread, flame retardants prevent the combustion process. The most essential chemical process about combustion can be realized in the vapor and/or

condensed phase. Combustion is suppressed by interrupting the exothermic processes. Physical means can inhibit the combustion process. Gas concentration produced by a flame retardant such as hydrogen bromide is sufficiently high; an inert gas, like nitrogen or carbon dioxide, may give a suppressant effect as a physical mechanism (Tkar, 1981).

The substrate is additionally cooled by using hydrated fillers as they decompose; for sustained pyrolysis, the temperature is lowered below what is needed. Decomposition of the polymer can be speeded up with the incorporation of an additive, such as peroxide, and this additive causes dripping of the polymer or pronounced flow. Removal of heat from the flame zone is achieved by this dripping of flaming polymer and dripping of flaming polymer essentially leads to flame extinguishment (Handani et al., 2009; Liang et al., 2013). Another part of additives is composed of the nanoparticles of metal oxide, silica, and polyhedral oligomeric silsesquioxane. These nanoscale particulate additives are determined by their sizes and have furthermore been verified to be very exciting on account of flame-retardant polymer systems (Metehen and Mustafov, 2017).

2. Materials and Methods

2.1. Materials

The materials used include retort stand, razor blade, lighter, stop watch, magnetic stirrer JB-4A, and analytical weighing balance JT 2003A.

2.1.1. Collection of PS wastes

PS waste samples were collected from a refuse dump site located at latitude 10°17′57″N and longitude 9°50′06″E Jahun, Bauchi State Nigeria.

2.1.2. Chemicals and reagents

In this work or research, the nanoparticles of NiO, BaO, and Sb_2O_3 used were synthesized in the Laboratory of Department of Chemistry, Abubakar Tafawa Balewa University, Bauchi. The solvents and reagents used were obtained in the laboratory and were of great analytical reagent grade, such as ethanol (Emerk Darmstadt Company), toluene (BDH Laboratory Reagent), methanol (Emerk Darmstadt Company), sodium hydroxide (Kermel), sodium bicarbonate (Kermel Company), nickel sulfate (AR Guandong Chemicals), barium nitrate (JHD Company), and antimony trichloride (Tited Biotech Ltd).

2.2. Methods

2.2.1. Dissolution of PS

The PS wastes were rinsed off and washed several times with detergent and water to remove any food or dirt particles and dried in the laboratory. The samples (PS wastes) were crushed so that they can be fit into the container. The PS waste (4 g) was dissolved in 20 cm^3 of toluene (solvent) in a conical flask and stirred for 30 minutes using a magnetic stirrer of model JB-4A. The solution was then allowed to be heated for 30 minutes at 60°C to form a solution (Achilias et al., 2015).

2.2.2. Preparation of nanoparticles

2.2.2.1. Preparation of BaO nanoparticles

In this research, a wet chemical precipitation method was used to obtain the metal oxide nanopaticles. Sodium bicarbonate (0.4 M) was added to the 0.2 M solution of barium nitrate drop wise, under constant stirring for 2 hours until addition of sodium bicarbonate solution was complete. After the completion of the reaction, the precipitates formed were allowed to settle overnight. The solution was filtered and washed several times with distilled water until free from excess bicarbonate. A white precipitate formed was then allowed to dry in oven at 80°C for 1 hour and then calcined at 150°C for 3 hours to form the desired barium oxide nanoparticles (Prabhavathi et al., 2014).

2.2.2.2. Preparation of NiO nanoparticles

A solution of nickel sulfate (1.0 L, 0.11 M) was taken and aqueous ammonia was added drop wise with constant stirring using a magnetic stirrer of model JB-4A until pH 10 was reached. The precipitate formed was filtered and washed several times with distilled water. The precipitate formed was allowed to dry in oven at 70°C for 24 hours and calcined at 600°C in a muffle furnace for 5 hours to form the desired nickel oxide nanoparticles (Vivek et al., 2014).

2.2.2.3. Preparation of Sb2O3 nanoparticles

A hydrothermal method was used to obtain Sb_2O_3 nanoparticles. 0.1 M solution of antimony trichloride was dissolved in 500 cm^3 of ethylene glycol under vigorous stirring to form a transparent solution. Subsequently, 500 cm^3 of distilled water was added to the solution and stirred for 15 minutes, and 6 M solution of sodium hydroxide (NaOH) was added to adjust

the pH. The whole solution was stirred for 20 minutes before transferring it into an autoclave at 120°C. After 12 hours, the product formed was centrifuged and washed several times with distilled water and ethanol and then dried at 60°C for 6 hours to obtained the required antimony oxide nanoparticles (Sb_2O_3) (Chen et al., 2008).

2.2.3. Formation of nanocomposites

In this experiment, an *in situ* polymerization method according to Edreese (2016) was used to obtain the desired nanocomposites. The nanoparticles of varying concentrations of 50, 100, 150, 200, and 250 mg for nickel oxide, barium oxide, and antimony trioxide were incorporated separately into the polymer solution of constant volume of 10 cm^3 and stirred for 1 hour using a magnetic stirrer of model JB-4A. The mixture was maintained at 60°C for 20 hours to promote *in situ* polymerization. The products formed were poured into excess methanol and stirred for 15–20 minutes and washed several times with ethanol and water. The products formed were oven-dried at 80°C overnight to obtain the desired nanocomposites in the form of a sheet. The sheet of the formulated nanocomposite of length 6 cm and width 3 cm were cut off using a razor blade.

2.3. Flame test

Each of the prepared nanocomposite was held in a position by a tong which was clamped to a retort stand and ignited using a lighter at the base and at the same time a stop watch was activated. The time taken for the combustion was recorded. The procedure was repeated for nanocomposites containing barium oxide, nickel oxide, and antimony trioxide nanoparticles. The following parameters were measured: ease of ignitability, rate of flame propagation, after glow time, and time taken for the combustion (Boryo et al., 2010, 2013).

2.3.1. Determination of ignitability

Ignition time is the time delay before the fire lighting. This was obtained by bringing light source in contact with the base of the nanocomposites. Ignition time was taken when the ignition source came in contact with the sample and became lightened. The sample was clamped vertically to a retort stand in a room with no air current and ignited at the base (Boryo et al., 2010, 2013).

The procedure was repeated for formulated nanocomposites containing nickel oxide, barium oxide, and antimony trioxide nanoparticles.

2.3.2. Determination of flame propagation rate (FPR)

The method described by Boryo et al. (2010, 2013) was adopted. The length of the formulated nanocomposite was taken before and after burning. The time taken for combustion was also taken and the distance travelled by flame was determined by subtracting the final length from the initial length. The FPR was determined using the following formula:

$$\text{FPR} = \frac{\text{Distance travelled by flame (cm)}}{\text{Time (second)}}$$

The procedure was repeated for formulated nanocomposites containing nickel oxide, barium oxide, and antimony trioxide nanoparticles.

2.3.3. Determination of afterglow time

The afterglow time was taken when the formulated nanocomposites continued to glow under a specified test condition after the cessation of the flame using a stop watch (Boryo et al., 2013).

3. Results and Discussion

3.1. Ignitibility of formulated nanocomposites

The result showing the effect of nanocomposites on ignitability is shown in Figure 1.

The result in Figure 1 shows that the untreated PS took 2 seconds before it ignites.

From Figure 1, it can be observed that the ignitibility of PS/Sb_2O_3 nanocomposite increases from 2 to 6 seconds with increasing Sb_2O_3 nanoparticles' concentration. It can also be observed from Figure 1 that PS/BaO nanocomposite showed an increase in the ignitibility from 2 to 5 seconds as the concentration of BaO nanoparticles increased. The nanocomposite containing NiO nanoparticles (PS/NiO) showed an increase in the ignitibility with increasing concentration, as shown in Figure 1.

The overall result clearly showed that ignitibility increases with an increasing concentration of nanoparticles. The nanocomposite containing Sb_2O_3 nanoparticles gave a significant fire retarding effect on ignitibility, followed by nanocomposites containing BaO and NiO nanoparticles. This may be due to the fact that Sb as a metalloid has some metallic and non-metallic properties, while Ba is an alkali earth metal (light metal) and Ni a transition metal. This fact is ascertain since the nanocomposites properties are reported by researchers to be directly dependent on matrix type (PS), filler-type (Sb_2O_3, NiO and BaO), interaction between the filler and polymer matrix, as

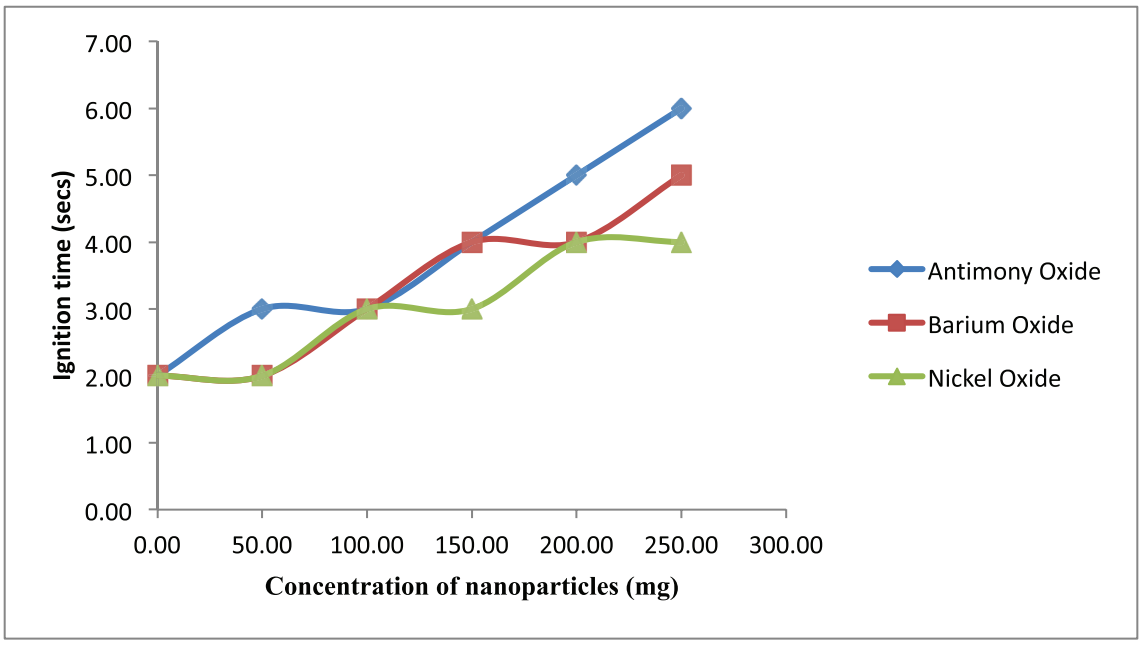

Figure 1. Effect of varying concentrations of nanoparticles on ignition time of formulated nanocomposite.

well as the distribution of the filler within the polymer matrix (Boryo, 2021; Nan et al., 1997; Wang et al., 2014).

This result conforms to the result obtained by Boryo et al. (2010, 2013) that as the concentration of the fire retardants, such as urea, borax, and ammonium chloride, increased, the ignitibility also increased (i.e., it took a longer time before the formulated nanocomposites ignited). It conforms to the result obtained by Kausar (2017) that the continuous reduction in peak heat release was recorded while incorporating C_{60} (Fullerene) in the polyurethane-coated poly(methyl methcrylate) (PMMA) matrix and compared to neat PU/PMMA, 0.5 wt% of Fullerene (C_{60}) added displayed 61% of reduce peak heat release rate. The Fullerene C_{60} reinforced composites showed prolonged time delay to ignition. It also conforms to the result obtained by Song et al. (2008) that when compared to untreated polypropylene, different amounts of C_{60} reinforced polypropylene composites have showed significantly reduced flammability rate and extended time to ignition.

3.2. Flame propagation rate of formulated nanocomposites

The result showing the effect of nanocomposites on flame propagation rate is shown in Figure 2.

From Figure 2, it was observed that untreated polystyrene has a flame propagation rate of 2.3 cm/sec. The result in Figure 2 clearly revealed that increase in the concentration of nanoparticles resulted to a slight decrease in the flame propagation rate for each of the formulated nanocomposites. It also showed that at higher concentration of each nanoparticles (that is 250 mg) the nanocomposites containing Sb_2O_3 and BaO (1.82 cm/sec and 1.80 cm/sec respectively) nanoparticles nearly compete in showing a better retarding effect on flame propagation rate.

The overall result showed that PS/NiO (1.53 cm/second) nanocomposite gave the best fire retarding effect on flame propagation rate followed by PS/BaO (1.80 cm/second) nanocomposite and then PS/Sb2O3 (1.82 cm/second) nanocomposite as shown in Figure 2.

This is because the lower the flame propagation rate, the less burnt in a given time and vice-versa.

This result conforms to the result obtained by Boryo et al. (2010, 2013). According to Boryo et al. (2010, 2013) urea gave a better fire retarding effect than borax and ammonium chloride as the concentration of fire retardants increased in ceiling board and PVC plastic. It also conforms to the result obtained by Song et al. (2008) that as compared to neat polypropylene, different amounts of C60 reinforced polypropylene composites have showed significantly reduced flammability rate and extended time to ignition.

3.3. Afterglow of formulated nanocomposites

The result showing the effect of nanocomposites on afterglow is shown in Figure 3.

From Figure 3, it can be observed that untreated PS shows an afterglow time of 5 seconds.It can be observed that an increase in concentration of Sb_2O_3

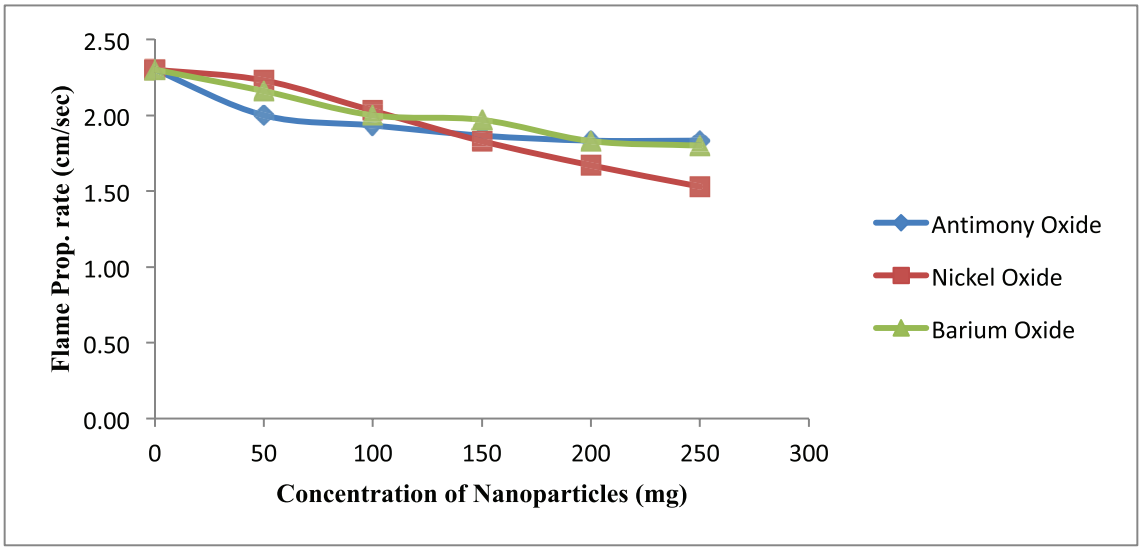

Figure 2. Effect of concentration of nanoparticles on FPR of formulated nanocomposites.

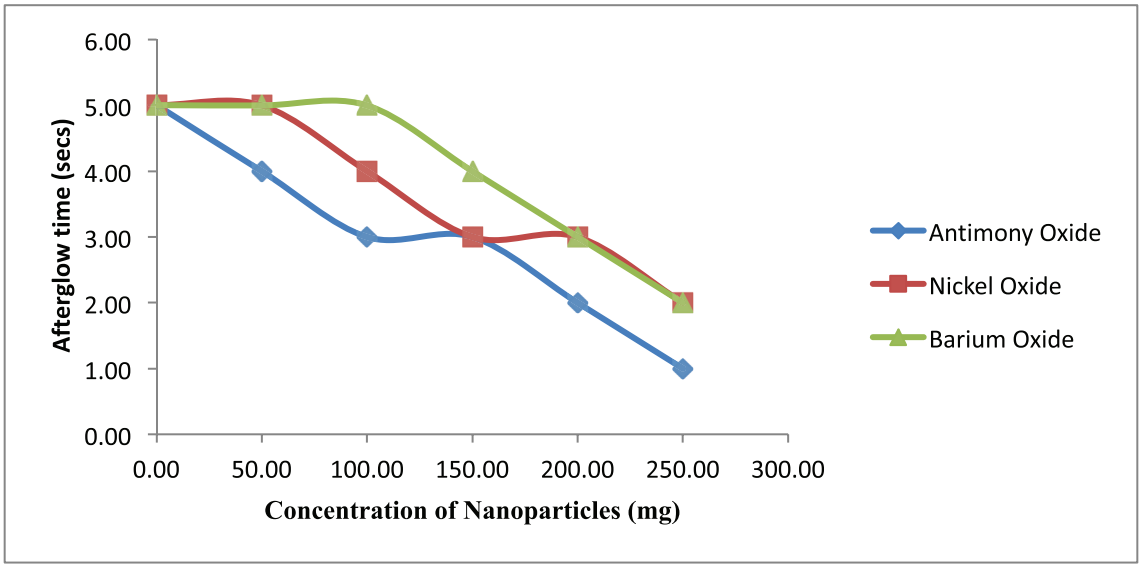

Figure 3. Effect of concentration of nanoparticles on afterglow time of formulated nanocomposites.

nanoparticle led to a decrease in the afterglow time of nanocomposite containing Sb_2O_3 nanoparticles. From the result in Figure 3, it showed that the afterglow time for nanocomposites containing NiO and BaO nanoparticles decreased with an increase in concentration and at higher nanoparticle content (250 mg), the PS/NiO and PS/BaO nanocomposites were competing in showing a fire retarding effect on the afterglow time.

From the figure, it can be observed that nanocomposite containing Sb_2O_3 nanoparticles gave the most considerable improvement on afterglow, while nanocomposite containing BaO nanoparticles gave more considerable and nanocomposite containing NiO nanoparticles as considerable. This result conforms to the result obtained by Boryo et al. (2013). According to Boryo et al. (2013), an increase in concentration of fire retardants (urea, borax, and ammonium chloride) yields a decrease in afterglow time but urea showed the most considerable improvement than borax which is more considerable and ammonium chloride as considerable.

3.4. Burning rate of formulated nanocomposites

The result showing the effects of nanocomposites on burning rate is shown in Table 1.

From Table 1, it can be observed that the burning rate of untreated PS was 0.0127 g. It showed that as the concentration of the nanocomposites containing NiO nanoparticles increased, and the burning rate decreased from 0.0128 to 0.0109 g/second.

Table 1. Effects of nanocomposites on burning rate

Weight of nanoparticle	Burning rate of nanocomposites (g/second)			
Incorporated into PS (g)	Blank PS	PS/NiO	PS/BaO	PS/Sb_2O_3
0.0	0.0127	-	-	-
0.05	-	0.0128	0.0112	0.0143
0.10	-	0.0118	0.0109	0.0139
0.15	-	0.0112	0.0103	0.0133
0.20	-	0.0112	0.0099	0.0131
0.25	-	0.0109	0.0098	0.0128

The burning rate for PS/BaO nanocomposite decreased from 0.0112 to 0.0098 g/second as the concentration increased. For nanocomposites containing Sb_2O_3 nanoparticle, the burning rate also decreased from 0.0143 to 0.0128 g/second with an increase in the concentration of nanoparticles. Considering the weight of the nanocomposite burnt in 1 second for PS/NiO, PS/BaO, and PS/Sb_2O_3 nanocomposites, PS/NiO nanocomposite had 0.0109 g burnt in 1 second, PS/BaO nanocomposite had 0.0098 g burnt in 1 second, and PS/Sb_2O_3 nanocomposite had 0.0128 g burnt in 1 second. This result shows that PS/Sb_2O_3 creates more char on combustion due to its synergistic effect but at low concentration of nanoparticles, the nanocomposite containing NiO nanoparticles gave a better retarding effect on burning rate due to the behavior of Ni as transition metal (heavy metal). This result conforms to the result obtained by Boryo et al. (2010, 2013) that ammonium phosphate gave more char than urea and borax as the concentration of retardants increased.

4. Conclusion

The nanoparticles were prepared via chemical precipitation method and the nanocomposites were fabricated using the *in situ* method of polymerization and characterized by various techniques. It was observed that PS/NiO, PS/BaO, and PS/Sb_2O_3 nanocomposites have fire retarding properties which increase as the concentration of the nanoparticles incorporated into the polymer matrix increase. It can also be concluded that PS/Sb_2O_3 nanocomposite has better fire retarding properties than PS/BaO and PS/NiO nanocomposites. It is recommended that during the production of polymer nanocomposites, PS/Sb_2O_3, PS/BaO, and PS/NiO nanocomposites can be used to hinder or slow down the propagation of fire during accidental fire outbreaks.

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