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EVALUATION OF FIREWORKS RESIDUE OBTAINED FROM THE FLASH POWDER CONTAINING ALUMINIUM AND BORON

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ABSTRACT

Herein, we report the characteristics of residues obtained after igniting the flash powder mixture consisting of varying amounts of Aluminium (Al) and Boron (B) with other energetic materials like potassium nitrate (KNO_3) and sulphur (S). The nature of particulate matter collected after combustion was characterized through FESEM-EDX, FTIR, STA (TGA/DSC) and ICP spectrometer. FTIR spectra analysis reveals about the presence of various metal oxides, K_2S and K_2SO_4. On replacing aluminum by boron, more alkaline K_2S present in the residue was converted into the neutral K_2SO_4. FESEM with EDX confirm the presence of the elements like Al, B, K, S, O, etc., and the size of the particles formed after combustion is about 400 nm. The gradual replacement of aluminum by boron is substantiated from the analysis of residue through ICP Spectrometer. Boron blended fireworks mixture on ignition produces excellent performances and the residues generated do not have any hazardous nature.

KEY WORDS : Aluminium, Boron, Flash powder, Fireworks, Residue.

INTRODUCTION

Worldwide, so many much of pollutants are released into the environment owing to the exorbitant use of various types of fuels in industries, automobiles, etc. Besides the gaseous and particulate pollutants released during burning the fossil fuels, more often particulate and gaseous pollutants in large amounts are released by the public while celebrating rituals, domestic and political functions. Annually, 60 metric tons of firecrackers were manufactured and burnt seasonally. The occasions like France's - Bastille Day, U.K.'s - Guy Fawkes Night, India's - Diwali, China and Taiwan's - Lantern Festival, Canada Day, etc. has been celebrated by bursting huge amount of firecrackers. Even though firecracker units generate more revenues, it always associated with a lot of risk factors. The unawareness about the usage of hazardous materials and improper disposal of waste is a significant source of pollution and also associated with a lot of health issues. While bursting firecrackers, it is obvious that the process releases too much amount of gaseous and particulate type of pollutants in large amounts. To eliminate the gaseous types of pollutants like CO_2, SO_x, NO_x, etc., researchers carried out their research on various types of alternatives (Ji *et al.*, 2018; Yao *et al.*, 2012). However, the particulates released during firecrackers display is not properly collected and analyzed. Furthermore, the particulate matters released during combustion are usually taken away from the bursting site owing to the flow of air currents.

In fact, the settling of the particulates may also induce some harmful effects to the croplands, living beings, building structures, etc., since; the burnt materials are normally in micron size and are readily dispersed in air and easily blown off until they reached the surface, where they cause the consequences. The dispersed fine particles cause negative health implications like coughing, wheezing, asthma, and even heart attacks. The oxides of the combustion products like Al_2O_3, K_2S, B_2O_3, etc. also have toxic effects, the same formed during celebrations are not reported anywhere. So, the analysis of the waste generated during the ignition of crackers is very important. Wilson *et al*. (2002) monitored the particulate matter in the outdoor space and classified them based on the particle size. Gozzi *et al.* (2016) suggested an easy way of monitoring particulate matter on a regional and local scale using mobile devices. Ismail (2017) reported the effects of nutrients present in the edible products after analyzing the ash obtained from combustion. The Environmental Protection Agency (EPA) of USA proposed steps to find out whether the ash collected from the burning of municipal wastes releases hazardous or toxic substances (6 EPA, 1995). In this context, numerous researchers reported about the characterization of the combustion residues of many materials, but the residues collected from fireworks wastes were never reported. These facts motivate us to collect the residues from fireworks and their characterization using FESEM-EDX, TG, FTIR and ICP. Conventionally, the raw materials used for making the firecrackers like Aluminium powder, potassium nitrate, ammonium nitrate and various color producing constituents like $Ba(NO_3)_2$, $Sr(NO_3)_2$, etc., produces various gaseous pollutants and particulate matters. Some important types of particulate matters produced after the combustion are alumina (Al_2O_3), K_2O, BaO, SrO, etc. Among the various types of products formed, some of them might be toxic, harmful to the environment. Flash powder composition consisting of potassium nitrate, aluminium and sulphur are the ingredients used to prepare almost all types of crackers. So, the residues of flash powder composition are collected after burning the firecrackers in a closed condition. Recently, we reported about the usage of boron as an alternative to the more hazardous aluminium and the boron blended fireworks composition display many remarkable characteristics like high stability, less sensitive to impact and frictional loads, etc. Besides, the more advantageous characteristics, in this paper, we reported the characteristics of residues obtained after burning the boron blended flash powders. Also, pH study of the residue was also conducted to find the impact of the soil quality as it would affect the availability of plant nutrients.

METHODOLOGY

Flash powder composition is made by mixing potassium nitrate (KNO_3), aluminium (Al), and sulphur (S) at the proportions of 57%, 23%, and 20% respectively based on the composition suggested by the Government of India (Explosive Rules 2008). The chemicals used are procured from the local suppliers in Sivakasi, Tamil Nadu, India. The mixture is sieved for three to five times to obtain a homogeneous mixture. From this composition, the proportion of aluminium is gradually replaced with boron in five different steps. Open burning tests are conducted as per the standard mentioned in the Environmental Protection Agency (EPA, 1993). The 60 x 60 cm porcelain tile is taken, and the sample is kept on the middle of the tile (approximately 7 cm diameter area). A nichrome wire of 30cm length is assembled in such a way that the wire is made to touch the sample followed by connecting to the electrical power supply.

The mixture is ignited by giving a power of about 102 watts for a moment, immediately the flash powder mixture catches fire and the left out the residue present over the surface of the tiles are collected. The collected ashes are analyzed through FESEM (ZEISS, Germany) to detect the size of the particle, FTIR (Bruker, Alpha, Germany) to identify the vibrational frequencies existing between the constituents present in the residue and ICP (Perkin Elmer US, Optima 7000 DV) to quantify the respective heavy metal elements present in the residue after burning.

Preparation of Samples

The flash powder composition consisting of KNO_3, S, Al and B prepared in five different combinations (B1 to B5). In these five combinations, the percentage of KNO_3 and S are kept constant as 57% and 20% respectively. The percentage of Al and B are varied are as follows: B1 contains 23% Al only, B2 contains 20.7% Al & 2.3% B, B3 contains 11.5% Al & 11.5% B, B4 contains 2.3% Al & 20.7% B and B5 contains 23% B only.

Collection of Residues

After igniting the flash powder mixture, the residues are collected as per the ASTM Standard [SW-846: Hazardous waste test methods]. 30 g of the sample is burnt in the porcelain tile using 30 cm Nichrome wire. The switched mode power supply (SMPS) capable of producing 12V DC with 8.5 Amps rating is used as the ignition power source. During ignition, the propagation of flame is photographed using a digital camera; the photographic images are given in Figure 1.

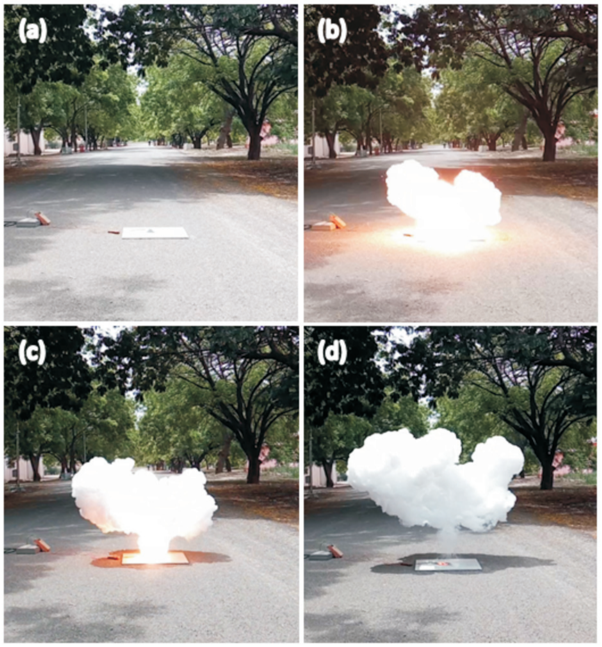


Fig. 1(a-d). Combustion stages of open burning test for Sample 1

Fig. 1(a-d) clearly depicts the progress of ignition from starting to end of the combustion process. During the combustion process, the parameters like flame height and time required to finish the combustion are observed. It is observed that, by increasing the quantity of boron, the height of the flame and the time taken for the combustion is found to be increased from 0.15 m to 0.3 m and 3 seconds to 6 seconds respectively.

The combustion characteristics of the mixture are analyzed from the following equations (1). Flash powder mixture consisting of KNO_3, Al & S (B1) undergoes combustion in presence of atmospheric air.

$$2KNO_3 + S + 4Al \rightarrow 2Al_2O_3 + K_2S + N_2- \quad .. (1)$$

During the combustion process, solid products like alumina (Al_2O_3) and potassium sulphide (K_2S) are formed and the gaseous product (N_2) is escaped out (Equation 1). From sample B2 onwards, the % of boron is gradually increased whereas the % of Al is decreased. Owing to the presence of boron, it also burns in air, produces its own oxide B_2O_3 (boron trioxide) along with other components as per the equation (2). Equation 3 represents the combustion of boron with other constituents since it contains only B, so it produces boron trioxide instead of Al_2O_3.

$$4KNO_3 + 2S + 4Al + 5B \rightarrow 2Al_2O_3 + 2B_2O_3 + 2K_2S + 2N_2 \quad .. (2)$$

$$2KNO_3 + S + 2B \rightarrow 2B_2O_3 + K_2S + N_2 \quad .. (3)$$

All these equations support the fact that, after burning, the solid products like Al_2O_3, K_2S, B_2O_3, left out as residues in a finely divided state, can be easily blown in the air. From the literature, it is noted that K_2S gets oxidized under higher temperature forming K_2SO_4 (Kosanke *et al*., 1996) owing to the higher exothermic nature of boron, it is expected that the boron blended mixtures may produce K_2SO_4 as shown in equation 4.

$$K_2S + 2O_2 \rightarrow K_2SO_4 + \text{Heat} \quad .. (4)$$

DSC analysis

The mixtures obtained viz. B1 to B5 are subjected to differential scanning calorimetric (DSC) analysis to determine the enthalpy associated during the combustion process. The analysis was carried out in N_2 atmosphere at the heating rate of 20°C/min. The heat of enthalpy liberated for samples B1 to B5 is found to be 1265 J/g, 1276 J/g, 1884 J/g, 1923 J/g and 2320 J/g respectively. From these DSC data, it is observed that the heat released during the process is increased. Such an increase in enthalpy is attributed to the increase in the quantity of boron, since in contrast to Al, B is having higher enthalpy (Liu *et al.*, 2013). Flame height obtained during burning is also differing due to the replacement of Al by the B content. Similarly, the time taken for the completion of burning the mixture also follows the same trend. All these results clearly reveal that the enhanced amount of boron present in the mixture exhibit excellent performance characteristics as required by the fireworks.

Characterization of the Residues

FESEM

FESEM analysis of the residues collected after burning, reveals about the size and shape of the particles obtained. The FESEM image with EDX was obtained from ZEISS, Germanyis shown in Fig. 2 (a&b). Fig. 2a depicts the FESEM image of residue collected from sample B1. FESEM image of the particles collected from sample B5 is shown in Fig. 2b.

From Fig. 2 (a&b), it is clear that the residues obtained from the combustion of the samples containing Al alone and B alone are different in size. FESEM image of the residue of the sample which contains Aluminium only shows uniform spherical particles which are having mostly uniform size of

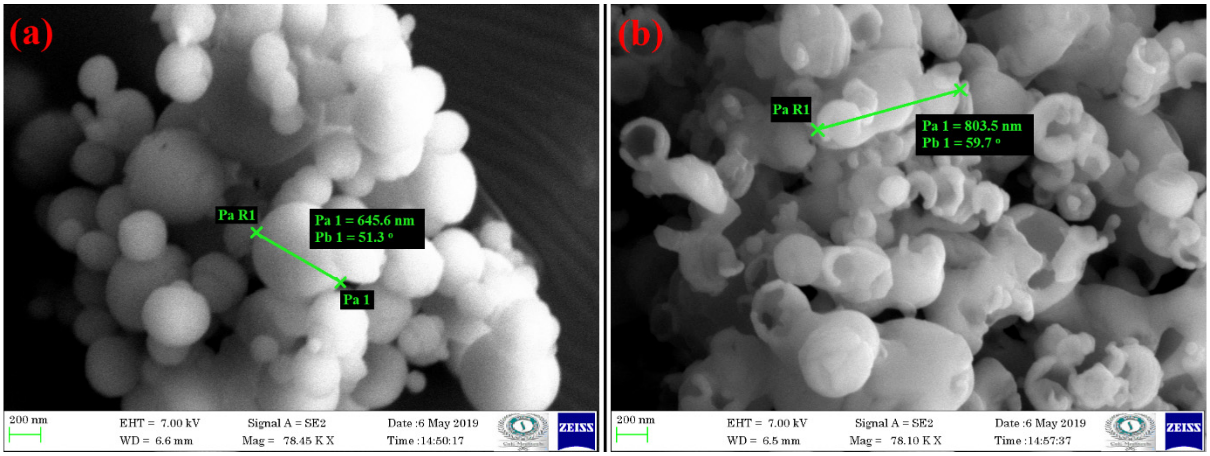


Fig. 2. FESEM images for the residues from a) Sample contains Al only and b) Sample contains B only

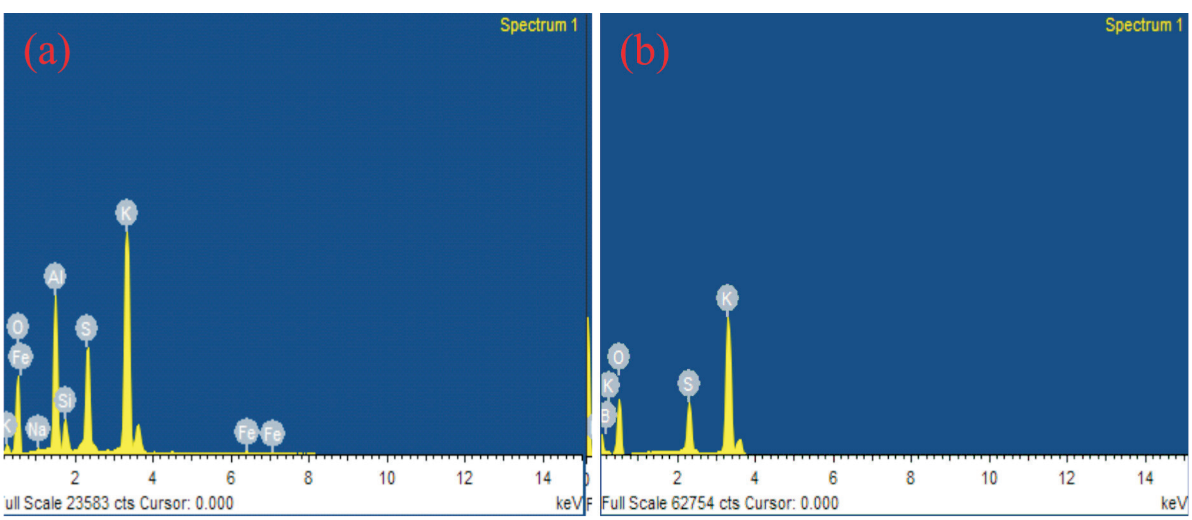
645 nm. Also, from the image, it is evident that these spherical particles are not formed a lump and easy to disperse in the air. Hence, particulate matter $PM_{2.5}$ will be increased to create air pollution. But, in the image of the residue of Boron contained sample, particles are not of uniform size and shape. The particle size has been increased and form a lump sized one. Hence, the weight of the particles had been increased and tends to settle on the floor, not tends to disperse in the air.

Fig. 3 (a&b) shows the EDX images of the residues collected from samples B1 (contains Al alone) and B5 (contains B alone). It exhibits the characteristic peaks for the presence of S, K, Al & B. The EDX data clearly reveals the presence of Al (Fig. 3a) with other constituents like S, K & O. Whereas Fig. 3b display the peaks for B substantiate that the mixture (B5) contains no Al.

FTIR

To validate the chemical constituents present in the ash, the FTIR spectra of all the samples are recorded in 500 – 4000 cm^{-1} region and are depicted in Fig. 4 (a-e).

Fig. 4 shows only prominent peaks around 3500 – 3600cm^{-1}attributable to the–OH vibrational peak, which are originated due to the adsorption of atmospheric moisture on the surface of the ash formed during combustion. Another peak appeared at 2300$cm^{-1}$is characteristic to the adsorbed carbon dioxide peak (Prathna *et al.*, 2018) existing as a carbonate. The FTIR spectra of boron blended composition (B3 – B5) after burning do not exhibit any peaks corresponding to the adsorption of



(a) (b)

Fig. 3. EDX images of after confined burning for a) Sample contains Al only and b) Sample contains B only

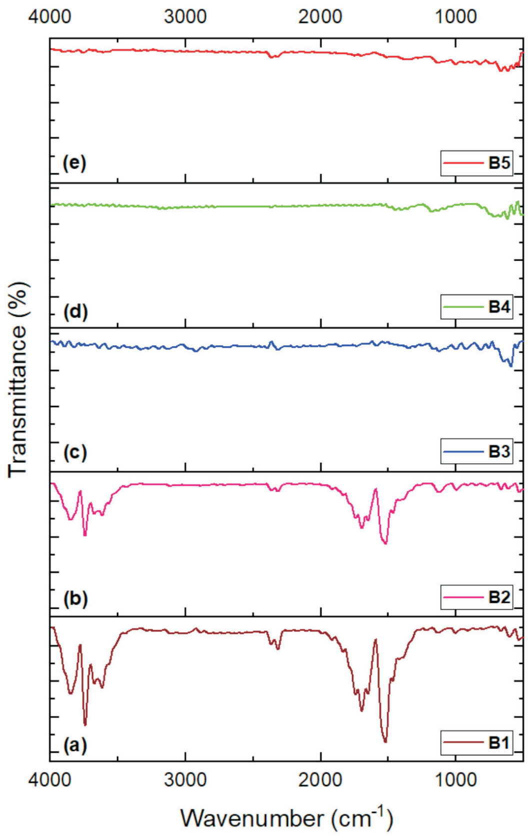


Fig. 4. FTIR spectra of samples collected after burning a) B1, b) B2, c) B3, d) B4 and e) B5

atmospheric moisture and CO_2 on their surface, it exhibits only the peaks corresponding to metal – oxygen vibrational peaks like B-O, Al-O in the lower wave number (500 – 600 cm^{-1}) regions.

To validate the absorbed moisture and CO_2 on the ash content obtained after combustion, the ash content collected was subjected to Thermo gravimetric analysis (TGA), Perkin Elmer, TGA4000, USA, at a heating rate of 20°C/min in N_2 atmosphere is shown in Fig. 5.

From the graph, it is evident that a slight loss in weight percentage of the mass of the residue is noticed at 100 °C characteristic to the loss of 2.5%, which is attributable to the loss of moisture associated with the specimen (Guo *et al.*, 2006). Another noticeable mass change occurred at 200 – 250 °C with a weight loss of 10% is ascribed to the escaping of CO_2 (Guo *et al.*, 2006). The mass loss associated at 100 °C and 200 – 250 °C confirmed the existence of moisture and CO_2on the residues collected. Adsorption of CO_2 & H_2O remains on the left out are already proved from the FTIR data too.

The FTIR spectra of all residues display the

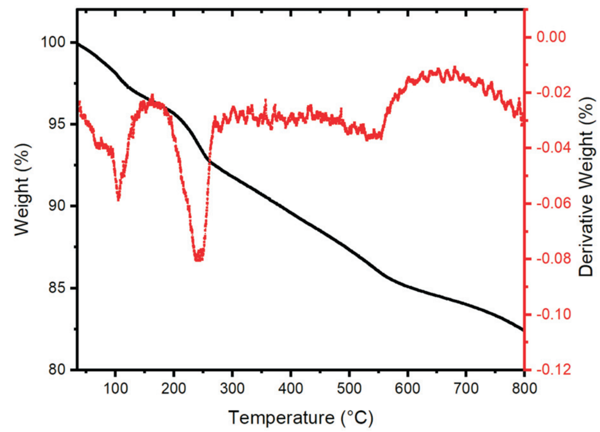


Fig. 5. TGA analysis of the residues of the sample 1 (contains Al only)

characteristic vibrational frequencies for both CO_2 and H_2O. To avoid such an adsorption over the residues, the collected ashes were heated at about 200 °C for 1 hour. The sample obtained after heating was immediately subjected to FTIR analysis and shown in Fig. 6. Heating at 200 °C facilitate the removal of both H_2O and CO_2 present in the specimen (Guo *et al.*, 2006). While heating the specimen at higher temperature, the possibility of adsorption of atmospheric moisture and CO_2 is totally restricted. So the FTIR specimen not show any characteristic vibrational peaks attributable to –

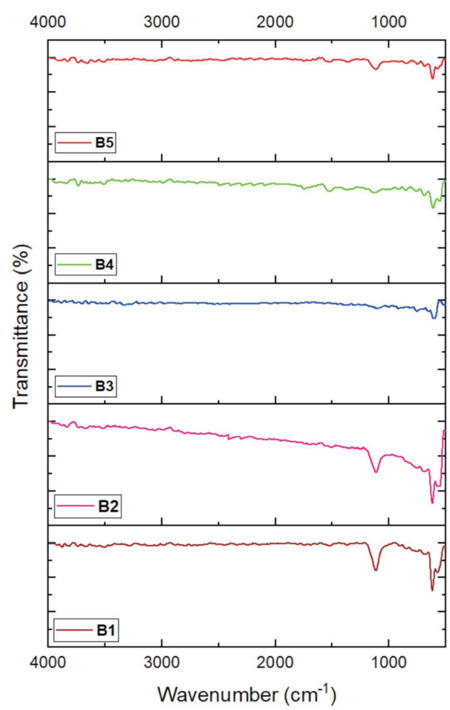


Fig. 6. FTIR spectra ofthe samples after heating

OH & carbonate variety. Since the ashes contains only metal oxides like B_2O_3, Al_2O_3 and the K_2S, their vibration frequencies are appeared only in the lower wave number region.

Since the spectra do not reveal any characteristic vibrational frequencies above 1500 cm^{-1}, the expanded spectra in the region 500 – 1500 cm^{-1} are depicted in Fig. 7.

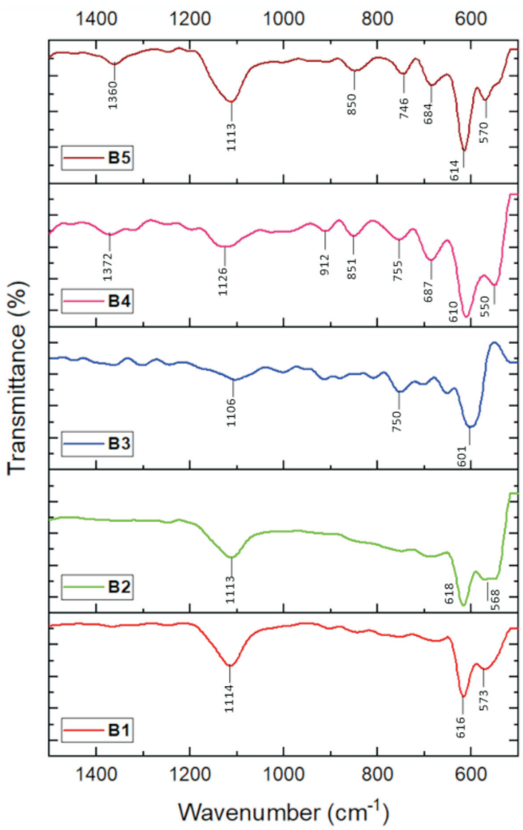


Fig. 7. Enlarged viewof the FTIR for the samples obtained after heating

FTIR spectra of the specimen obtained in the lower wave number region exhibit a prominent vibrational band characteristic to the presence of K_2S on all the samples at about 1120 cm^{-1} (Tosun *et al.*, 2013).The peak exists at around 620 $cm^{-1}$and 570 cm^{-1} is originated from the Al_2O_3 (Benykhlef *et al.*, 2016; Djebaili *et al.*, 2015) present in the ash. While reducing the quantity of Al by replacing with boron, the intensity of peak found at 1120 cm^{-1} gradually decreases. The peak originating at 685 cm^{-1} and 1350 cm^{-1} is due to the presence of B_2O_3 content in ash (Kumar *et al.*, 2015). All these vibrations have appeared only on those samples having higher % of B, i.e. B3 – B5.

Regarding the explosive mixture B1, it mainly consists of Al with others, favors the formation of K_2S as a solid material with Al_2O_3. While reducing Al, the quantity of B is increased from B2 to B5. On increasing B, besides the formation of B_2O_3, it also facilitates the formation of K_2SO_4. Presence of K_2SO_4 along with the ash contents are noticed from the FTIR spectra (Periasamy *et al.*, 2009). From the literature data, vibration of K_2SO_4 would be asymmetric bending at the frequency of 612.8 cm^{-1}. Here, in these spectra of B4 and B5, peak formed in 610 and 614. Also, small peaks were appeared in the range of 1285.7 cm^{-1} in B3 to B5, which is matched with the literature data of K_2SO_4 vibration due to S-O-H stretching and bending of HSO4 group.

When comparing K_2S and K_2SO_4,K_2S is flammable, highly corrosive and toxic, and it may also induce dust explosion in the finely divided state (Pub Chem, 2022; WHO, 2000). In contrast to K_2S, K_2SO_4 is nonflammable but decomposed when exposed to heat. K_2SO_4 lies in the green circle (safe chemicals) listed out by the United States Environmental Protection Agency (20 EPA, 2019) through a safer choice program. From these results, it is inferred that when boron is added with fireworks mixtures, the corrosive and toxic nature of the ash content is greatly reduced. It is also noted that non-flammable products are formed if the fireworks mixture consists of higher % of B. From the result, it is ensured that the residues formed after combustion is non-toxic and non-hazardous.

pH Test

To know the reactivity of the ash particles obtained with environment moisture, the pH of the aqueous solution was monitored by suspending known quantity of residues in water. The pH values of the samples were measured by suspending 1 gram of ash in 100 ml of water. It is found that the pH values of all the samples are almost the same which is equal to 8.50 (base). From the FTIR results, it is clear that the different by-products are formed during burning. On burning B1, produces mostly Al_2O_3 and K_2S, which on mixing with water, produces a basic effect since in water K_2S produces potassium hydroxide (KOH) and potassium hydrogen sulphide (KHS) (21 Ncchemist, 2022). Since the ash dispersed suspension containing KHS, KOH (formed from K_2S) and Al_2O_3, the pH of the suspension is reduced to 8.5.

Whereas in the samples B2 to B5, the quantity of Al is gradually replaced by an increasing amount of B. From the FTIR results, it is confirmed that the formation of K_2SO_4 is increased when boron is added to the mixture. Therefore, the quantity of K_2S produced is reduced, which makes the liquid less basic when compared to that of B1. Hence, when the quantity of boron is increased, the amount of K_2S formed is also reduced, which decreases the alkalinity of the solution gradually. But, the quantity of Al is also reduced, hence the tendency to reduce the pH is also very less. On the whole, the K_2S (basic) and Al_2O_3 (acidic) are gradually reduced, which makes the pH value to be uniform. At the same time, the oxide of boron forms a weak acid (boric acid) when dissolved in water and the K_2SO_4 acts as neutral (HSBD, 2019) in water and hence the role of B_2O_3 and K_2SO_4 can be neglected. Therefore, by blending boron to the fireworks mixture, decreases the hazardous nature. So, after combustion, the ash will not cause any corrosive effect and 8.5 pH value will not affect the environment as much because even the groundwater systems have the pH value lying in the range of 6 – 8.5 (Oram, Brian, 2022).

ICP Results

The ashes collected after burning from the samples B1 to B5 are subjected to Inductively Coupled Plasma Spectrometer (ICP) analysis. The ICP analysis of the samples (B1-B5) dissolved in aqua-regia was injected after proper dilution. The elemental constituents like aluminium and boron are analyzed; the obtained results are given in Table 1.

Table 1. ICP results for the residues obtained

Sample Name	Aluminium (mg/l)	Boron (mg/l)
B1	45.80	0
B2	41.09	4.52
B3	23.08	23.06
B4	4.48	41.50
B5	0	45.68

The data clearly reveals that the % of Al decreases from B1 to B5. Similar results in the increasing order with reference to boron are noticed. As the samples are prepared by decreasing the composition of Al (23% to 0%) simultaneously increasing the boron from 0% to 23%. The ash contents having their corresponding stoichiometric composition of Al as Al_2O_3 and B as B_2O_3, in all the compositions the % of Al and B are present in almost double the percentage. Since during ignition, both Al & B are converted into their own oxides in the format M_2O_3. By dissolving the residue (100 mg) in aqua regia, the % of metal concentration is doubled. Potassium is found almost similar in all the samples. Any other toxic metals like chromium, lead, cadmium, etc. are not found in this ash.

CONCLUSION

The residues left over after the combustion or detonation of explosives blended with boron were analyzed and compared with the results of aluminium blended mixtures. Characterization techniques like FESEM-EDX, FTIR, and ICP were used for analyzing the residues. The FESEM image shows that the size of the particle is increased when boron is added, thereby reducing the particulate matter formation. The FTIR results show that the peak denoting the presence of K_2S is reduced indicating the formation of K_2SO_4 when boron is present in the mixture. ICP results also confirm that the increase in boron content is found in residues. The boron present in the composition influences the conversion of flammable, corrosive and toxic components like K_2S into a non-flammable inert K_2SO_4. Further, it also reduces particulate matter formation. On the usage of boron blended firecrackers, it is possible to eliminate the risk associated with the particulate matters. Also, the soil quality with respect to pH also did not affect. The results obtained from these studies ensured that the hazardous nature of aluminium based fireworks is modified while replacing the aluminium with boron.

Conflict of interest: None

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