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Full Length Research Article

ENHANCING FIRE RETARDANCY IN POLYPROPYLENE COMPOSITES USING ALKALI-TREATED DATE SEED POWDER AND SbO_3: A STUDY OF THE EFFECTS OF PARTICLE SIZE

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ABSTRACT
This study investigates the fire-retardant (FR) properties of polypropylene (PP) composites reinforced with alkali-treated date seed powder (DSP) of variable particle sizes (63 µm and 750 µm). These properties are described under different concentrations of NaOH treatment (0% control, 1%, 5%, and 10%), alongside varying concentrations of FR (5% and 10%). This research evaluates the composite's thermal stability and flame-retardant performance by examining critical key parameters, including the limiting oxygen Index (LOI), ignition time and extent of burning. Results indicate that smaller particle sizes (63 µm) treated with a high concentration of NaOH (10%) exhibited better fire retardancy compared to larger particle sizes (750 µm), which demonstrated greater stability across all treatments because of uniform dispersion and char layer formation. The composite receiving 10% NaOH treatment and 10% FR achieved the highest LOI values of 20.71 for 63 µm DSP and 20.41 for 750 µm DSP, indicating a moderate level of flame resistance while highlighting the need for further optimisation for elevated fire safety standards. This study has revealed that employing bio-based fillers to enhance the flame retardancy of polymer composites represents a promising pathway for future sustainable materials in industries such as automotive, construction, and consumer goods. Other chemical treatments or further FR agents should be considered in future studies for improved fire resistance.

Keywords: Date Seed, Polypropylene Composites, Alkali Treatment, Fire Retardancy, Particle Size, Ignition Time

INTRODUCTION
Many manufacturing industries are concerned with fire safety, which has led to efforts to improve the flame-retardant properties of polymer composites. Among these polymers, polypropylene (PP) is unique for its availability, ease of processing, and good mechanical properties (Idumah, 2022). However, the end product's inherent flammability necessitates fire-resistant additives to curtail the incidence of fire hazards in environments sensitive to fire. Recently, natural fillers such as date seed powder (DSP) and fibres were used as sustainable substitutions for conventional additives, providing possible performance and environmental sustainability benefits (Li et al, 2025).
Incorporating date seed powder in the polypropylene matrices opens avenues to engendering bio-based composites of superior fire-retardant (FR) properties. Specific effective treatment techniques should be enhanced to improve the performance and compatibility of DSP within the polymer matrix, to obtain optimal FR performance (Jefferson et al., 2024). This has been studied previously to modify surface properties and interfacial interaction of natural fibres with polymer matrices employing chemical treatments like alkali treatment using sodium hydroxide (NaOH) (Lawal et al. 2023).
This paper examines the influence of selected NaOH concentrations (0%, 1%, 5%, and 10%) on the ignition time and extent of flame propagation in PP/DSP composites. It considers two particle sizes, 63 µm and 750 µm. To enhance the fire-retardant properties of the composites, a constant concentration of 5 % SbO_3 was added, as a recognised flame-retardant agent compatible with other additives (Ibrahim et al., 2024). This approach was designed to improve the fire-retardant effects while ensuring consistent fire retardancy assessment under the different NaOH treatments.
The Limiting Oxygen Index (LOI) is the minimum oxygen level required to sustain combustion and serves as an indicative metric for the efficiency of FR treatments. Therefore, LOI and other fire performance criteria like ignition time and burning extents will be used to assess the capability of the composite to slow flames and minimise combustibility. This study will explain the main processes that influence fire resistance in PP/DSP composites across various particle sizes and NaOH treatment levels. The findings will be applicable for understanding bio-based FR additives in polymer composites as potential long-term solutions for fire safety in sectors including automotive, construction, buildings and household items.

MATERIALS AND METHODS
Polypropylene was collected as waste, washed with detergent, thoroughly rinsed with water and then dried. Date seeds were obtained from Yan Dabino, Zaria City market of Kaduna State. Antimony trioxide (Sb_2O_3) is the fire retardant used in composite production. NaOH was used to improve bonding between the filler and matrix. All chemicals used in the experiments were of analytical reagent grade acquired from Romptech Chemical Company in Zaria, Nigeria, product originally supplied without further purification.

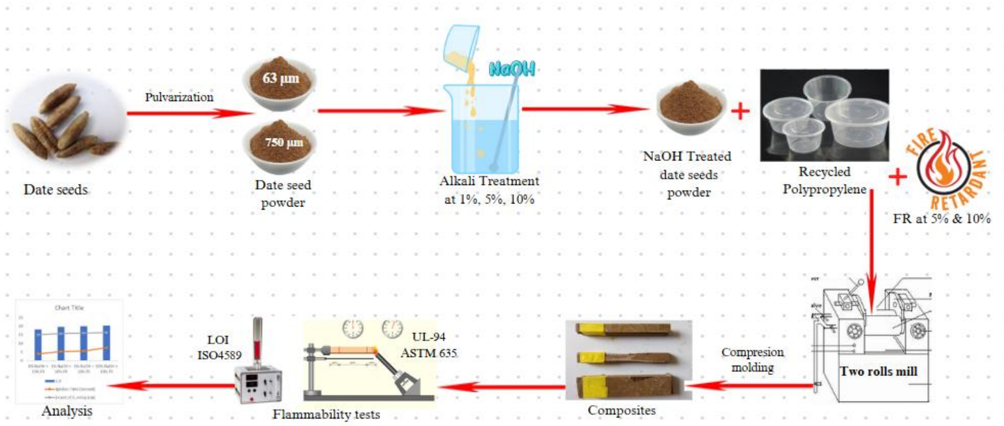

Figure 1: Polypropylene (PP) matrix integrated with date seed particles (DSP) of two sizes: 63 µm and 750 µm

Sample preparation

The cleaned and dried polypropylene (wPP) was cut into flakes and kept in an air-tight container for future work. Date seeds were first cleaned in water to remove impurities, then sun-dried before being ground using jaw crusher and ball mill machines (Retsch Masch. Nr 70992 GMbH and CO. and Kera b.v. Soeter Berg Overveld 057748 Holland, respectively). The ground date was then sieved using an Impact Lab Sieve (ISO 3310-1:2000, BS 410-1:2000) to obtain particle sizes of 63 µm and 750 µm. Subsequently, the particles underwent alkali treatment at 5% and 10% w/v concentrations by immersing them in a NaOH solution for 1 hour, at a ratio of 1:10 (date seed particles to NaOH solution). After treatment, the date seeds were washed several times in water until a neutral pH of 7 was achieved. It was finally allowed to dry in an oven at 70°C for 24 hours (Elnaid et al., 2020).

Fabrication of the composites

The composites were compounded and fabricated using compression moulding techniques at 20 % filler loading using a two-roll mill and compression moulding machines. A steel mould of 150 × 120 × 5 mm was used. The control sample was the unfilled waste polypropylene. The matrix and filler were mixed for 5 minutes in a two-roll mill to achieve a homogenous mixture. The mixture was placed in the mould, treated with a releasing agent. The maximum pressing temperature, pressure, time and cold pressing or pressure holding time were 160 ^{0}C, 5 N/mm^2, 15 minutes and 5 minutes, respectively (Lawal *et al*., 2019). After cooling, the composite was removed and conditioned in an oven at 70 ^{0}C for 48 hrs.

Table 1: Composition of PP/DSP Composite Samples

Sample	Particle Size (µm)	NaOH Treatment (%)	DSP Loading (wt.%)	FR Loading (wt.%)	PP Content (wt.%)
0% NaOH+10 FR_63	63	0	20	10	70
1% NaOH+10 FR_63	63	1	20	10	70
5% NaOH+10 FR_63	63	5	20	10	70
10% NaOH+10 FR_63	63	10	20	10	70
1% NaOH+5 FR_63	63	1	20	5	75
5% NaOH+5 FR_63	63	5	20	5	75
10% NaOH+5 FR_63	63	10	20	5	75
0% NaOH+10 FR_750	750	0	20	10	70
1% NaOH+10 FR_750	750	1	20	10	70
5% NaOH+10 FR_750	750	5	20	10	70
10% NaOH+10 FR_750	750	10	20	10	70
1% NaOH+5 FR_750	750	1	20	5	75
5% NaOH+5 FR_750	750	5	20	5	75
10% NaOH+5 FR_750	750	10	20	5	75

Flammability test

The flammability test (UL-94) was carried out following ASTM International D635. The samples in 125 × 13 × 3 mm were clamped horizontally at one end, and the burner was placed toward the other end for the flame to impinge on the free end. The time and extent of burning were measured for the flame that travelled from the 25 mm marking to 100 mm needed to be recorded. Subsequently, the rates of burning were calculated using equation (1) as follows:

$$V = \frac{60L}{t} \quad (1)$$

Where:

V is the rate of burning (mm/min), L is the burned length (mm), and t is the time of burning (sec).

LOI test

The LOI was determined using the oxygen index apparatus from Fire Testing Technology Limited, located in West Sussex, United Kingdom. The test was conducted using method 141 and ISO 4589

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with an inflow of 17 litres per minute at a temperature of 23 ± 2 °C, with Polypropylene composite dimensions measuring 90 x 6 x 3 mm^3.

RESULTS AND DISCUSSION

Flammability

Figure 2 - 5 illustrates the effect of different NaOH concentrations on the extent of burning in polypropylene/date seed powder (PP/DSP) composites at two different particle sizes: 63 µm and 750 µm. The extent of burning, expressed in millimeters, is used to assess the composites' flame-retardant properties. The study uses a consistent 5% FR concentration across varying levels of NaOH treatments (0%, 1%, 5%, 10%).

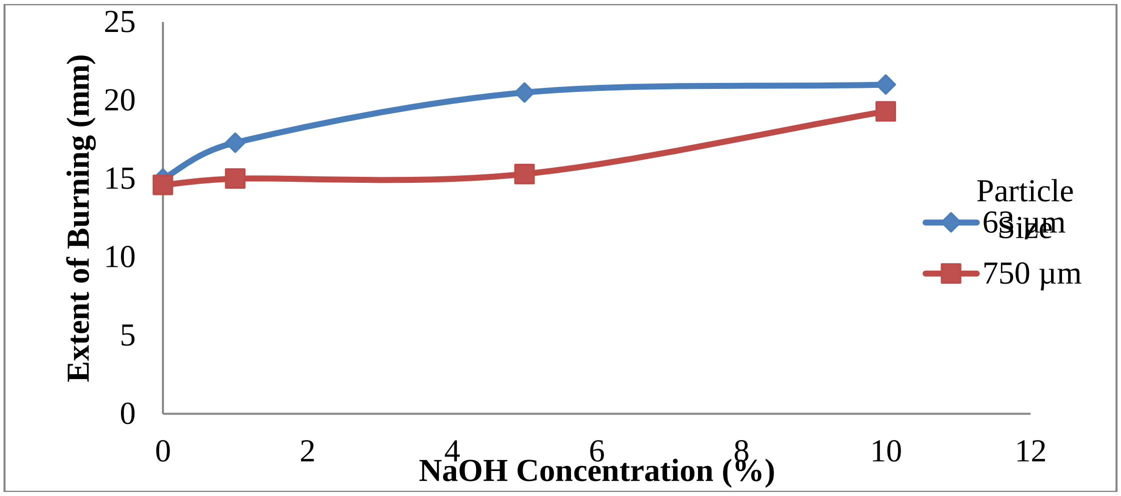

Figure 2: Effect of NaOH Concentration (%) on the Extent of Burning of PP/DSP Composites (5% FR) at Different Particle Sizes

Figure 2 illustrates how different concentrations of NaOH affect the burning extent of PP/DSP composites with particle sizes of 63 µm and 750 µm, at a consistent 5% flame retardant. It shows an increase in the extent of burning with higher NaOH concentrations for the 63 µm particles filled composite. At 1% NaOH, the burning is moderate, but it becomes significant at 10%. This indicates that higher alkali levels may reduce the effectiveness of the flame retardant by degrading the fibre surface or interfering with its interaction with the polymer matrix (Jefferson, 2024). Similar research has shown that alkali treatment weakens fibres and creates porosity, and spreads flames more easily (Mohammed et al., 2023).

In contrast, the 750 µm particles demonstrate greater stability, with only a slight increase in burning as the NaOH concentration rises. This stability may be due to better bonding between the matrix and the fibres, resulting in less surface degradation and enhanced flame resistance (Shesan et al.2019; Mohammed et al., 2022). Smaller particles (63 µm) do not show any benefits from higher alkali treatment, while larger particles (750 µm) maintain consistent flame-retardant properties. This finding aligns with previous research that highlights the influence of particle size and chemical treatment on flame retardancy (Jefferson et al., 2024).

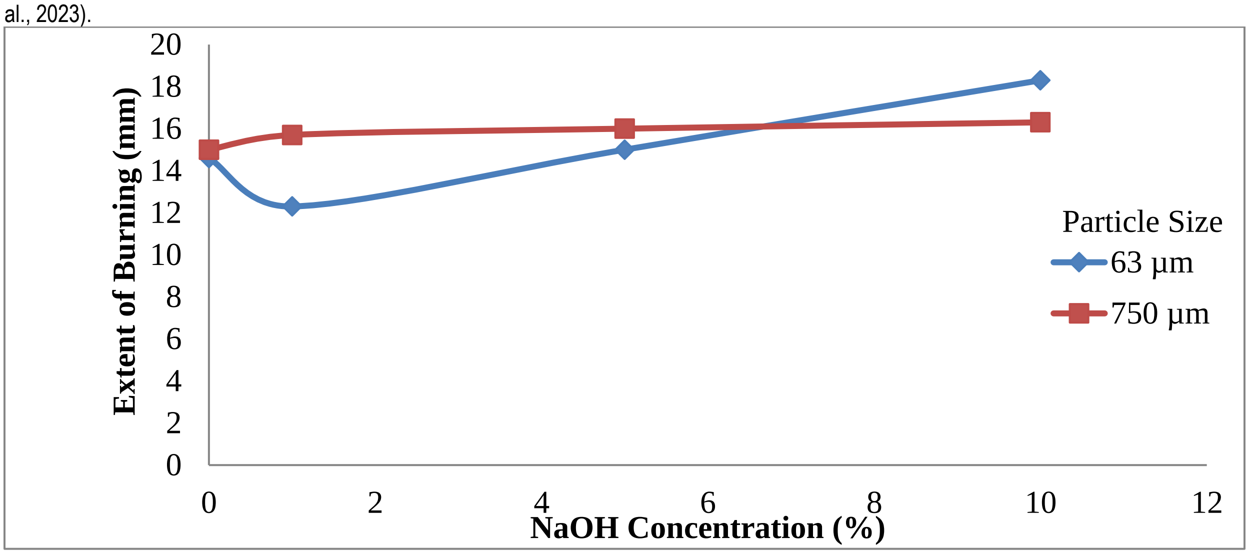

Figure 3: Effect of NaOH Conc. (%) on the Extent of burning of PP / DSP composites (10 % FR) at different particle sizes

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Figure 3 shows the impact of varying NaOH concentrations on the flame resistance of PP/DSP composites with particle sizes of 63 µm and 750 µm, using a 10% FR. For the 63 µm particles, the burning extent shows a notable curved trend:-at approximately 16 mm without NaOH, decreases to about 12.5 mm at 4% NaOH, and then rises to around 18 mm at 10% NaOH. This suggests that a moderate NaOH concentration enhances flame resistance by improving fibre-matrix bonding, but excessive concentrations may lead to fibre degradation (Amiandamhen et al., 2022).
In contrast, the 750 µm particles maintain a consistent burning extent of around 16 mm across all NaOH concentrations, indicating that larger particles interact uniformly with the matrix, thus preserving flame resistance. Similar research supports that larger fillers promote thermal stability and flame resistance (Rothon, 2003).
Moderate NaOH treatments for 63 µm particles yield optimal flame resistance, while excessive treatment harms it. The larger 750 µm particles are less affected, making them a more reliable option for stable flame-retardant performance. This study emphasises that optimising NaOH concentration based on particle size can enhance the flame retardancy of natural fibre-reinforced composites (Mahmood et al., 2024).

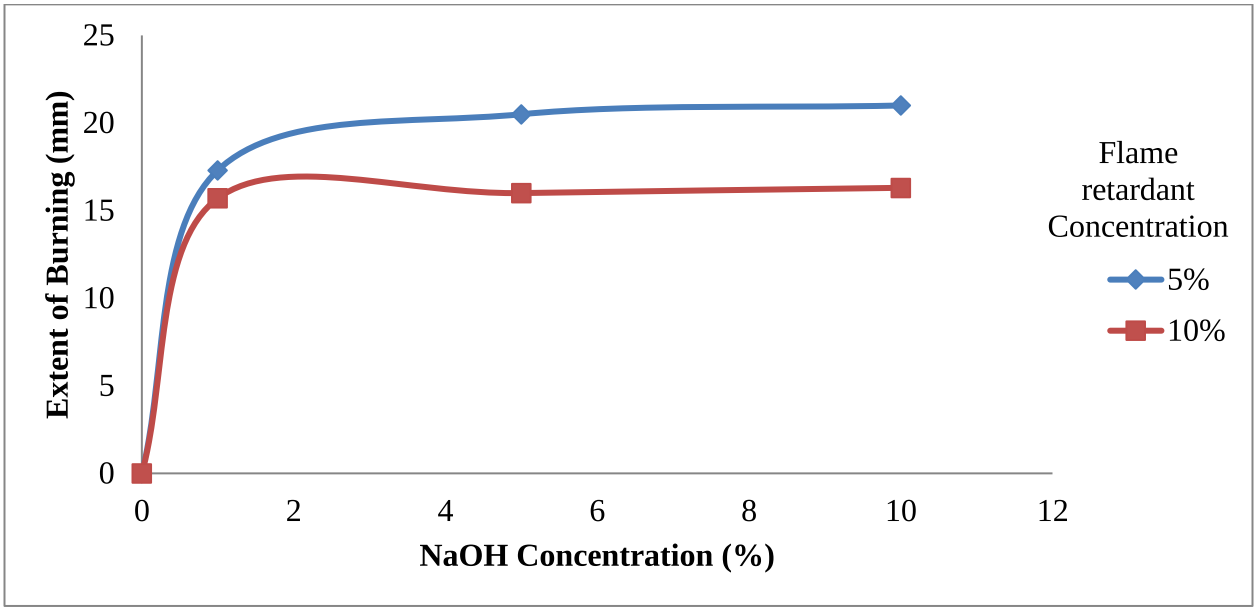

Figure 4: Effect of NaOH Conc. (%) on the Extent of burning of PP / 63 µm DSP composites at different flame-retardant concentrations

It was observed in Figure 3 that the burning extent increased from 0% to a NaOH concentration of 2%, ultimately reaching around 20 mm, for the composite with 5% FR. This suggests inadequate fibre modification occurs at lower NaOH concentrations, resulting in diminished flame resistance. Beyond the 2% NaOH threshold, the extent of burning remained relatively constant at approximately 21 mm, indicating no further improvement in flame resistance from higher alkali treatment (Alao et al., 2022; Chalapathi et al., 2022).
In contrast, the 10% FR composite exhibited a different behaviour. Initially, the burning extent was approximately 15 mm at 0% NaOH and showed a slight increase to just over 16 mm at 2% NaOH, after which it stabilised or slightly decreased with higher concentrations. The results indicate that the improved flame resistance due to higher FR content outweighs any advantages of more alkali treatment. This aligns with previous research suggesting that greater flame-retardant levels enhance thermal stability and lower flammability (Sivanantham et al.,2024).
The variation in flame retardant concentrations emphasises the importance of optimising NaOH and FR levels for effective flame resistance (Azwa et al. 2013). While the 5% FR composite demonstrated notable increases in the extent of burning at lower NaOH levels, the 10% FR composite displayed greater stability, highlighting a more effective interaction between the flame retardant and treated fibres (Amiandamhen et al., 2020).
Convincingly, composites with a higher flame retardant (10%) show enhanced flame resistance and stability across varying NaOH concentrations. On the other hand, the 5% FR composites are more sensitive to alkali treatment, supporting the observation that greater flame-retardant content contributes positively to the thermal stability and fire resistance of natural fibre-reinforced composites.

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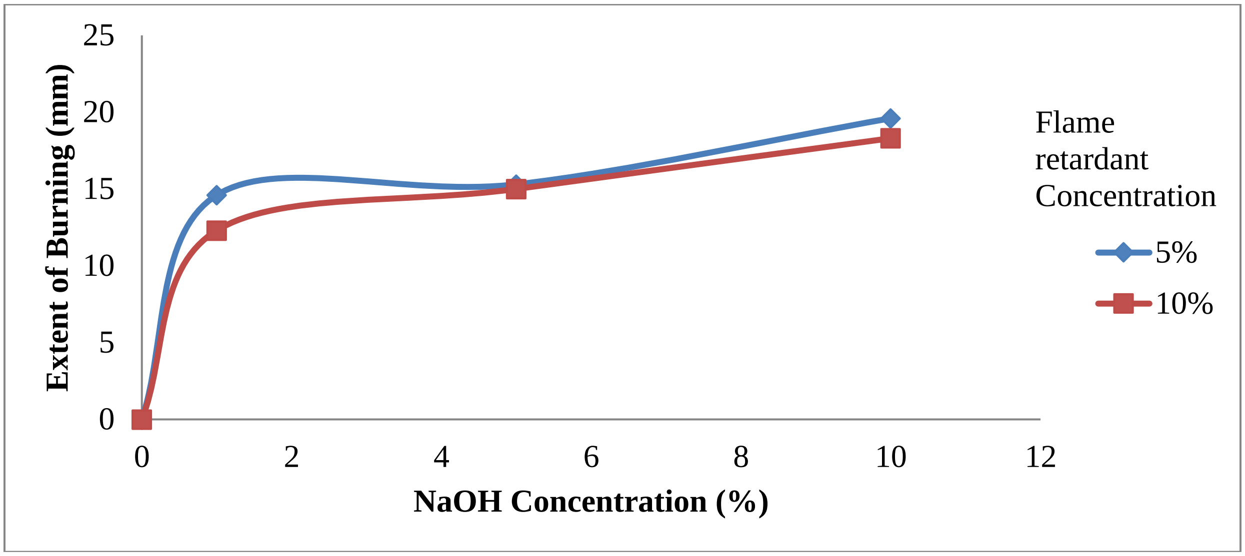

Figure 5: Effect of NaOH Conc. (%) on the Extent of burning of PP / 750 µm DSP composites at different flame-retardant concentrations

Figure 5 shows the effect of NaOH concentration on the burning extent of PP composites reinforced with 750 µm DSP at FR levels of 5% and 10%. For the 5% FR composite, burning increases from 5 mm to 17 mm as NaOH concentration rises from 0% to 2%, indicating significant flammability due to inadequate alkali treatment (Ahmadi et al., 2024). It then steadies and reaches about 21 mm at 10% NaOH.

The 10% FR composite follows a similar trend but exhibits lower burning extents, starting below 5 mm and peaking at approximately 20 mm. This suggests that a higher FR content improves flame resistance by forming a protective barrier during combustion (Shen et al., 2021). Higher NaOH levels generally lead to increased burning due to fibre degradation; however, the higher concentration of FR promotes the formation of a char layer (Madyaratri et al., 2022).

The difference in performance between the 5% and 10% FR composites highlights the importance of FR concentration. The 10% FR composite benefits from better synergy with the polymer matrix, while the larger particle size may affect dispersion, contributing to flammability. Generally, optimizing NaOH concentration and FR content is essential for developing effective flame-resistant PP/DSP composites (Ahmadi et al., 2024).

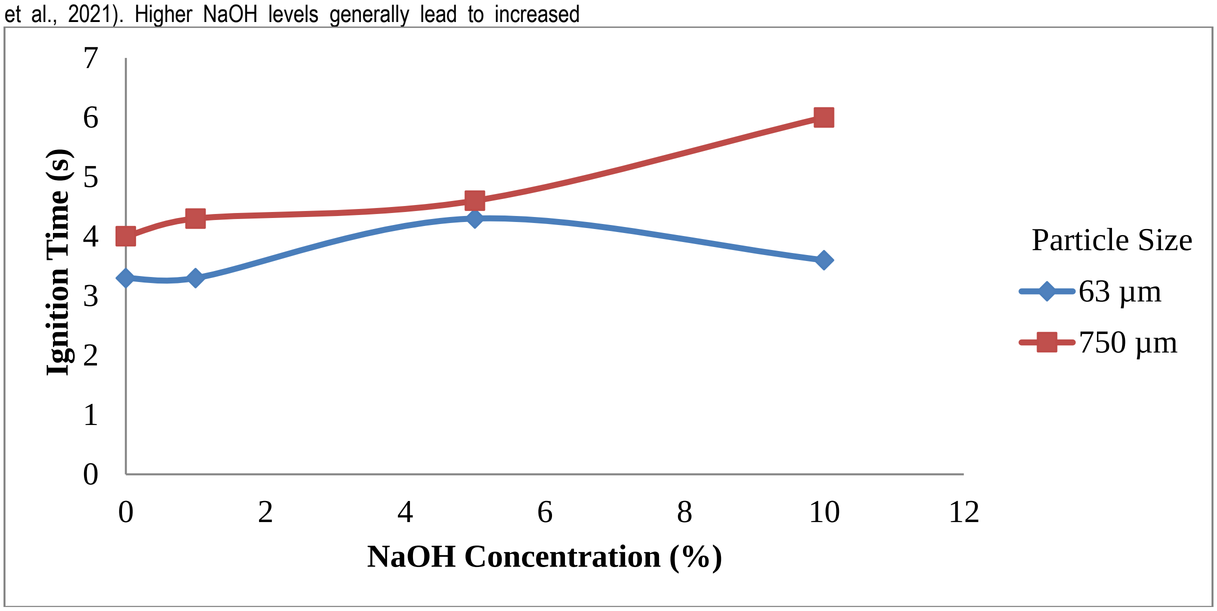

Figure 6: Effect of NaOH Conc. (%) on Ignition time (s) of PP / DSP composites (5 % FR) at different particle sizes

Figure 6 illustrates the influence of NaOH concentration on the ignition time of PP composites reinforced with DSP at a 5% FR concentration, using two particle sizes: 63 µm and 750 µm. Ignition time, measured in seconds, indicates how long it takes for the material to ignite when exposed to a flame, which is critical for assessing flame resistance.

For the 63 µm DSP composite, ignition time decreases from 3.5 seconds to 2.5 seconds as NaOH concentration increases from 0%

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to 2%. This suggests that low NaOH concentrations may cause surface degradation, reducing the particles' effectiveness in delaying ignition (Ahmadi et al., 2024). However, ignition time gradually increases, beyond this point reaching approximately 3.5 seconds at 10 % NaOH, indicating improved fibre-matrix adhesion at higher concentrations (Lee, 2018).

In contrast, the 750 µm DSP composite (red line) shows a steady increase in ignition time, rising from 4.5 seconds at 0% NaOH to around 6 seconds at 10% NaOH. The larger particle size enhances flame retardancy by contributing to a thicker char layer during combustion (Liu et al., 2022).

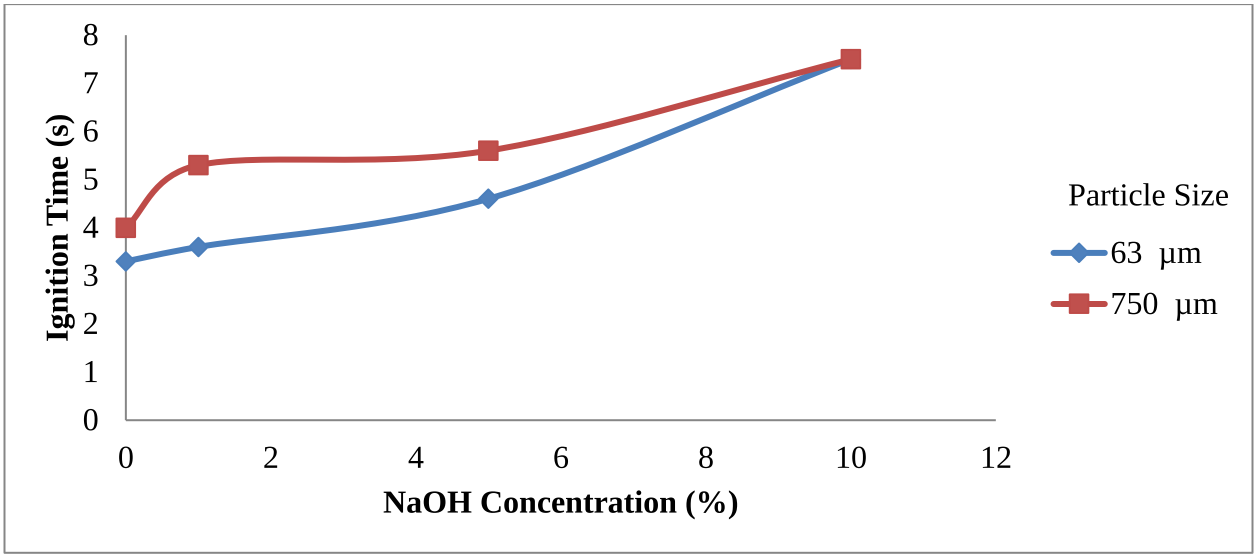

Figure 7: Effect of NaOH Conc. (%) on Ignition time (s) of PP / DSP composites (10 % FR) at different particle sizes

Figure 7 demonstrates the effect of NaOH concentration on the ignition time of PP composites reinforced with DSP at a fixed FR concentration of 10%, comparing particle sizes of 63 µm and 750 µm. The ignition time, which reflects the duration before the composite ignites, shows that the 63 µm DSP composite exhibits a steady increase from approximately 4 seconds at 0% NaOH to around 6.5 seconds at 10% NaOH, suggesting improved flame resistance due to enhanced bonding between the fibres and the polymer matrix (Bar *et al.*, 2015). In contrast, the 750 µm DSP composite starts at 4.5 seconds and reaches about 7 seconds at 10% NaOH, indicating that the larger particles provide better insulation and form a thicker char layer during combustion, delaying ignition.

These results highlight that both NaOH concentration and DSP particle size significantly impact ignition time, with the larger 750 µm particles outperforming the smaller ones. The 10% FR concentration enhances flame resistance in both composites, particularly at higher NaOH levels, indicating effective fibre-matrix bonding due to alkali treatment. Additionally, the finer 63 µm particles show a more pronounced increase in ignition time with increasing NaOH concentration, likely due to their greater surface area-to-volume ratio, making them more susceptible to surface modifications. In conclusion, Figure 6 emphasises the positive effect of NaOH concentration on the ignition time of PP/DSP composites and suggests that optimising particle size and alkali treatment is crucial for developing effective flame-resistant materials. Future studies could explore additional chemical treatments and higher flame-retardant concentrations to further enhance the ignition resistance of DSP-reinforced PP composites.

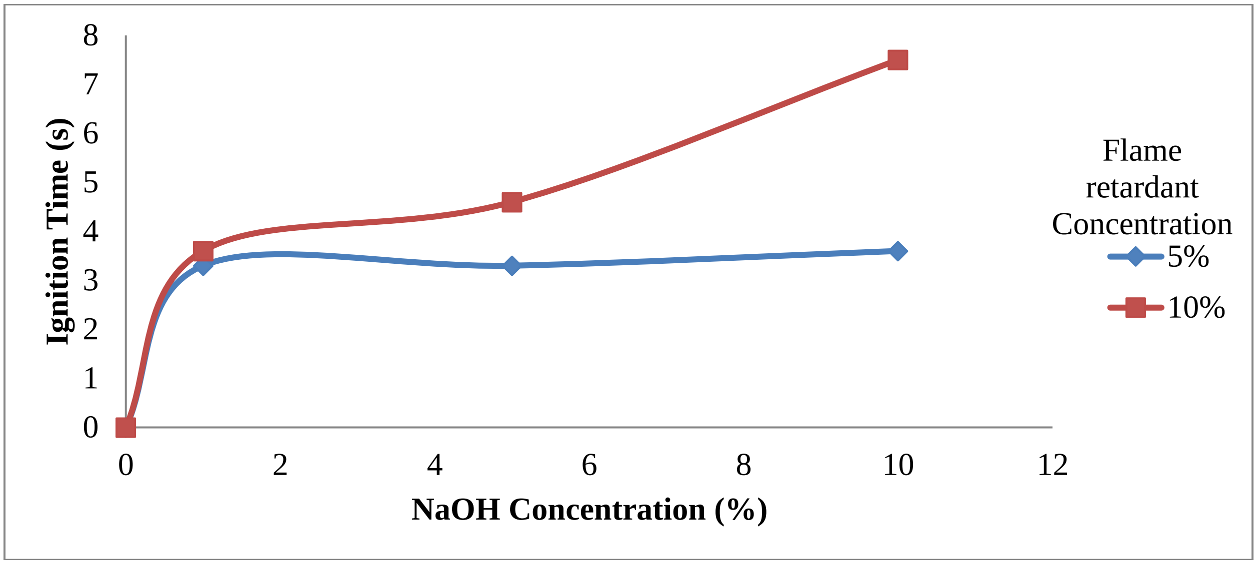

Figure 8: Effect of NaOH Conc. (%) on Ignition time (s) of PP / 63 µm DSP composites at different flame-retardant concentrations

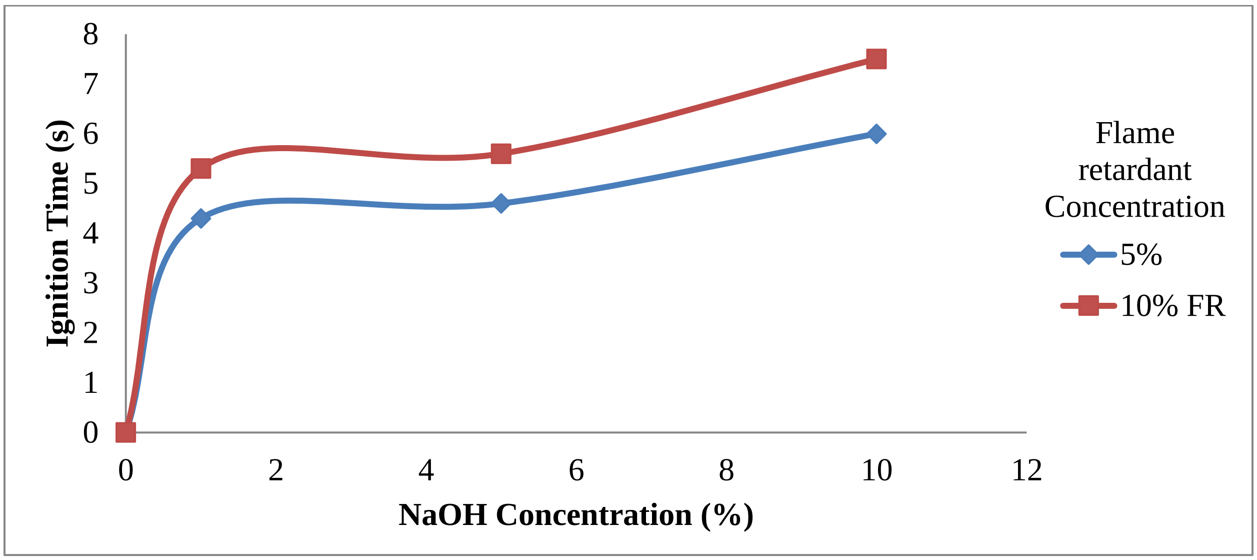

Figure 9: Effect of NaOH Conc. (%) on Ignition time (s) of PP / 750 µm DSP composites at different flame-retardant concentrations

The Limiting Oxygen Index

The LOI test for polypropylene/date seed composites provides insight into how particle size, alkali treatment concentration, and flame-retardant content affect the material's flame-retardant properties.

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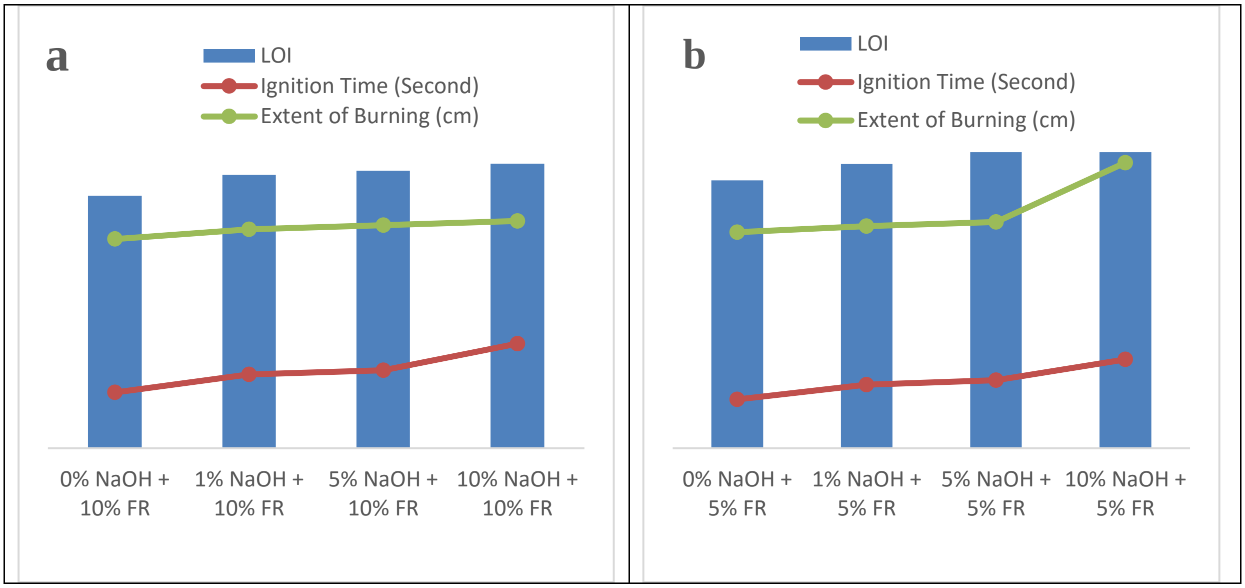

Figure 10: Influence of NaOH Treatment on the Fire Retardancy of PP/DSP Composites Containing (a) 10% and (b) 5% Flame Retardant

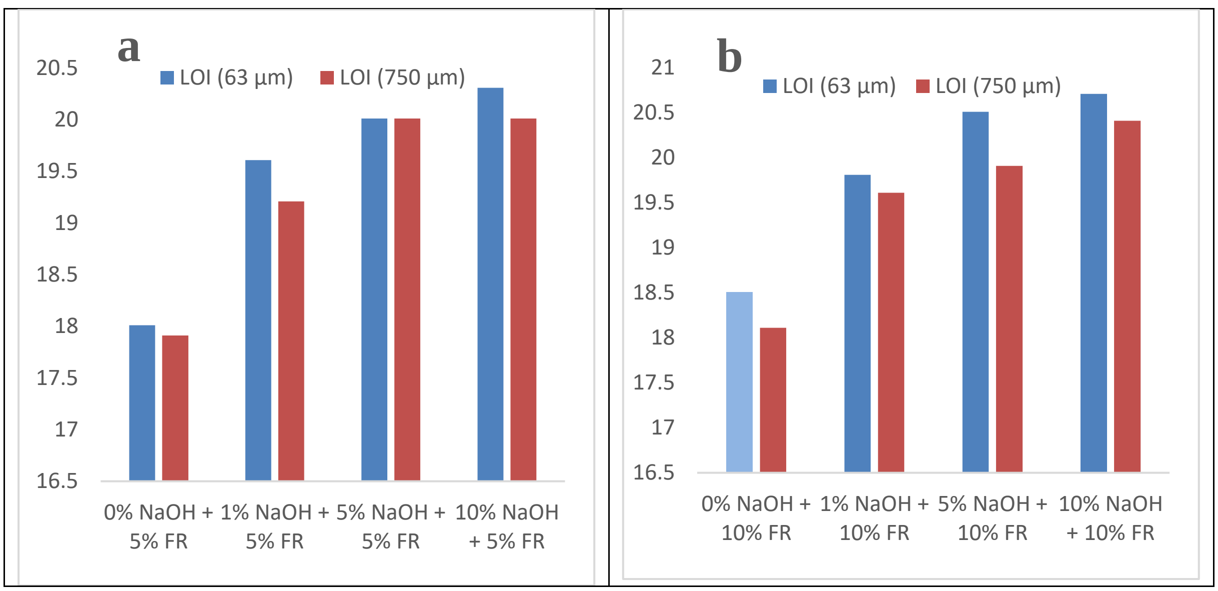

Figure 11: Influence of NaOH Treatment on the Limiting Oxygen Index (LOI) of PP/DSP Composites at Varying Particle Sizes with (a) 5% and (b) 10% Flame Retardant

Figures 11 show that finer particle sizes of 63 μm exhibited enhanced flame resistance, achieving a maximum LOI of 20.71 when treated with 10% NaOH and 10% flame retardant (FR). This indicates that smaller particles enhance the surface area interaction between the polymer matrix and the flame retardant, thereby improving the combustion resistance of the composite (Ganesan, 2025). On the other hand, composites with larger particle sizes, such as 750 μm, exhibited slightly lower LOI values (Irhayyim et al., 2025), reaching a peak at 20.41. This indicates that larger particles may reduce the effectiveness of the flame retardant due to decreased surface interaction and possible voids in the composite structure (Akindele, 2025).

The composites treated with a higher concentration of NaOH (10%) consistently exhibited better flame retardancy. The higher alkali concentration is likely to enhance fibre-matrix adhesion by removing impurities and increasing surface roughness, facilitating better bonding between the fibres and the flame retardant (Asyraf et al., 2025). In contrast, lower concentrations of NaOH, such as 1%, led to the lowest LOI values. This indicates inadequate fibre treatment and weaker interactions between the fibre and the matrix, resulting in diminished flame-retardant performance (Ganesan, 2025).

The concentration of flame retardant significantly impacts the material's flame resistance. A 10% FR concentration produced the highest Limiting Oxygen Index values, indicating that a higher amount of flame-retardant compounds in the matrix effectively delayed ignition and enhanced the composite's ability to resist combustion (Li X et al.,2025). In contrast, a 5% FR concentration

provided some flame retardancy, but its performance was comparatively lower. This highlights the importance of using a higher percentage of flame retardant for optimal effectiveness (Zhang et al., 2025).
The findings also indicate that the best flame-retardant properties were achieved with a particle size of 63 µm, a 10% NaOH treatment, and a 10% FR, leading to an LOI of 20.71. While this value reflects moderate flame resistance, it is not deemed excellent; materials with an LOI above 25 generally have good flame retardancy. Therefore, although these composites exhibited improved combustion resistance compared to untreated materials, they may still ignite under normal atmospheric conditions, containing approximately 21% oxygen. For applications requiring higher flame resistance, further optimization of the composite formulation or incorporating additional flame-retardant agents may be necessary.

Conclusion

This study investigated the impact of alkali treatment and particle size of DSP on the fire-retardant properties of PP composites, revealing that NaOH concentration, particle size, and FR content significantly influence flammability, thermal stability, and fire resistance. Smaller DSP particles (63 µm) treated with 10% NaOH and reinforced with 10% FR exhibited the highest Limiting Oxygen Index of 20.71 due to enhanced fibre-matrix bonding and dispersion. However, larger particles (750 µm) achieved a slightly lower peak LOI of 20.41 but demonstrated greater thermal stability across treatments through char layer formation. Excessive alkali treatment (above 5%) led to fibre degradation and reduced flame retardancy, particularly in smaller particles, while higher FR content (10%) consistently improved fire resistance. Although moderate flame resistance was achieved, the LOI values indicate that the composites may still ignite under atmospheric oxygen levels, necessitating further optimisation, such as using synergistic FR agents or advanced fibre treatments to meet stringent fire safety standards. The study concludes that DSP, as a sustainable filler, offers significant potential for enhancing the flame retardancy of PP composites in eco-conscious applications, with future research needed to optimise formulations and achieve superior fire resistance while maintaining environmental sustainability.

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