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The Effect of Di-Ammonium Phosphate (DAP) towards Flammability Properties of Polyester-Cotton Military Fabrics

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ABSTRACT

Polyester-cotton fabric is one of the most common materials used in military fabrics. The study of the suitable fabric material is critical because military personnel must prepare themselves from inevitable events such as heat exposure. Therefore, the purpose of this study was to examine the flammability features of various cotton compositions in polyester-cotton military textiles with and without the presence of Di-ammonium Phosphate (DAP). We performed a full flammability test, including a cone calorimeter test and a limited oxygen index (LOI) test on three distinct types of polyester-cotton textiles treated with 10% di-ammonium phosphate (DAP). The result shows that, the higher the percentage of cotton, the lower the fire-retardant properties. Besides that, this study also found that the di-ammonium phosphate (DAP) improves the flammable properties of the military fabric.

1. Introduction

Cotton fabric, crafted from organic cotton fibres through weaving, is highly favoured within the textile industry owing to its exceptional attributes of comfort, softness, and breathability. This fabric originates from the seed pod of the cotton plant, known as the cotton-boll, which contains fluffy fibres. However, it is essential to recognize that cotton-based products are susceptible to combustion, making cotton's flammability a matter of significant concern across diverse sectors, including automotive, electrical, hospitality, software, entertainment, food, and the chemical industry. Ahmed *et al.,* [1] said this flammability factor is especially critical not only for cotton clothing and home furnishings but also for cotton-based items utilized in various industrial applications.

To enhance specific attributes of cotton fabric, such as flexibility or durability, it is common practice to blend cotton with other fibers like polyester or spandex. These days, two distinct types of thread—natural/natural, natural/synthetic, or synthetic/synthetic—are always used for weaving

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fabric as stated by Razali *et al.,* [2]. Polyester stands out as one of the textile market's most flame-resistant materials due to its inherent resistance to ignition. Polyester, a synthetic polymer composed of polyethylene terephthalate (PET), exhibits excellent resistance to heat, shrinking, and mildew to its chemical composition, particularly the two monomers of ethylene glycol and terephthalic acid. Its molecular structure consists of tightly interwoven strands of molecules, and the strong hydrogen bonds formed between these molecules demand substantial energy to break, resulting in high heat resistance. This tightly woven structure contributes to the fabric's resistance to heat, making it a preferred choice in various applications as stated by Khan *et al.,* [4]. There are many approaches to improve fire properties of polyester/cotton blend such as by layer-by-layer coating with poly-(allylamine) and poly (sodium phosphate) [16], ammonium polyphosphate-/poly (acrylic acid) [17], phosphorus/nitrogen/silicon-containing nano-coating [18], ammonium polyphosphate-based coatings [19].

Diammonium Phosphate (DAP) serves as an effective intumescent fire retardant, well-suited for a wide range of polymers and polymer-based materials, including cotton. DAP's notable molecular weight, combined with its safety and cost-efficiency, makes it stand out in enhancing the Limiting Oxygen Index (LOI) of cotton. Its performance surpasses that of alternative phosphorus compounds like tributyl phosphate, triallyl phosphate, or triallyl phosphoric triamide as study by Putu *et al.,* [8]. It is well-established that flame retardants containing both phosphorus and nitrogen, derived from urea, exhibit superior effectiveness compared to single-element compounds.

The application using diammonium phosphate (DAP) to reduces the material's combustion temperature, increases the rate of weight loss during combustion, and generates substantial char volumes. DAP finds widespread use as a flame retardant, whether in a non-durable or semi-durable capacity, particularly for items subject to frequent use or intended for disposability. In line with specific research findings, DAP stands out as the non-durable and durable flame retardant that produces the most pronounced char formation and flame-retardant attributes in materials containing cellulose. The use of Diammonium Phosphate (DAP) as a flame-retardant additive was previously reported by Al-Taee *et al.,* [20], Veerappagounder *et al.,* [21] and Gaan and Sun [22].

In the context of our study, we investigated the flammability properties of polyester-cotton fabrics treated with DAP as a flame retardant. This study presents an initial exploration of employing the cone calorimeter for measuring fabric's heat release parameters. Additionally, our study delves into the impact of DAP on the Limited Oxygen Index (LOI), a critical factor in evaluating the flammability properties of polyester-cotton military fabric.

2. Methodology

2.1 Materials

In this study, the different composition of polyester-cotton fabric with the thickness 0.04 cm was used to investigate the flammability characteristics of fabrics. The details of composition of polyester-cotton fabric are given in Table 1. There are three (3) different composition of polyester-cotton military fabric with 10% of Di-ammonium Phosphate (DAP) used in this research. The size of each sample is prepared based on the test that done in this study.

Table 1
Composition of polyester-cotton Military Fabric

Sample	Percentage of Cotton	Percentage of Polyester
1	100 %	0 %
2	50 %	50 %
3	0 %	100 %

2.1.1 Sample preparation

All the sample is prepared based on the each of the test method where 10 cm X 10 cm for Cone Calorimeter Test and 120 mm X 50 mm for Limited Oxygen Index Test. Then the sample is emersed into a 10 % concentration of DAP for 1 hour. After that, the sample is dried under room temperature for 23 Hours before proceed for testing.

2.1 Methods
2.2.1 Cone calorimeter

In the testing of combination samples, a cone calorimeter operating on the principle of oxygen consumption, manufactured by Fire Testing Technology Limited in the UK, was employed. The testing procedure adhered to ISO 5660 standards. Fabric samples, measuring 100 mm x 100 mm, were meticulously cut from continuous sheets of various polyester-cotton fabric compositions. To ensure consistent and accurate testing conditions, all samples were affixed with a frame and grid, effectively securing the fabric flat and preventing any curling or movement that might lead to erroneous ignition results.

The testing setup involved enveloping a thermally insulating ceramic brick with a 4-inch (10.2 cm) width in aluminium foil. Subsequently, the 0.04 cm thickness fabric sample was positioned atop this aluminium-foil-wrapped brick. To maintain stability and uniformity during the test, the frame and grid were employed to securely hold all components in place. The fabric sample was positioned on a layer of aluminium foil and subjected to horizontal radiation under a heat flux of 50 kW/m^2 as mentioned by Cheng *et al.,* [3] and Liu *et al.,* [6].

2.2.2 Limited Oxygen Index

The determination of LOI (Limiting Oxygen Index) values followed the standardized ASTM D2863 method. An oxygen index apparatus from Fire Engineering & Science Technology (FESTEC) in Korea was employed for this purpose. The experimental specimen size used for LOI measurement was 120 mm x 50 mm, and it was securely fixed in a sample holder as per the testing protocol. To conduct the LOI test, the flow rates of nitrogen and oxygen were adjusted to the specified parameters. Subsequently, the fabric sample was ignited, and the resulting data, including the LOI value, was meticulously recorded. This method provides crucial information regarding the flammability and ignition characteristics of the fabric under controlled oxygen conditions. Previous researcher Liu *et al.,* [6] also used this test to expresses the lowest oxygen to nitrogen ratio where specimen combustion is still self-supporting.

3. Results
3.1 Cone Calorimeter

In this study, our primary focus was on two critical parameters: time to ignition (TTI) and peak heat release rate (PHRR), as illustrated in Figure 1 and Figure 2. TTI plays significant role in understanding the ignition behaviour of materials, including fabrics, as highlighted by previous research by Yang *et al.,* [15]. Fabrics with shorter TTI are more prone to igniting and spreading fires to their surroundings. TTI essentially measures how quickly a substance ignites and subsequently burns when exposed to heat as studied by Yang *et al.,* [15].

The introduction of flame-retardant agents had a positive impact on delaying the combustion process of polyester-cotton textiles. This is evident in the data presented in Figure 1, where fabrics with varying cotton percentages, ranging from 0% to 100%, showed reduced flammability. For instance, without Diammonium Phosphate (DAP), the ignition times ranged from 48 seconds for 0% cotton fabric to 8 seconds for 100% cotton fabric. However, when DAP was applied to the fabrics, the ignition times increased to 66 seconds for 0% cotton fabric and 12 seconds for 100% cotton fabric, indicating a substantial delay in ignition compared to fabrics without DAP as supported by study from Skorodumova *et al.,* [10].

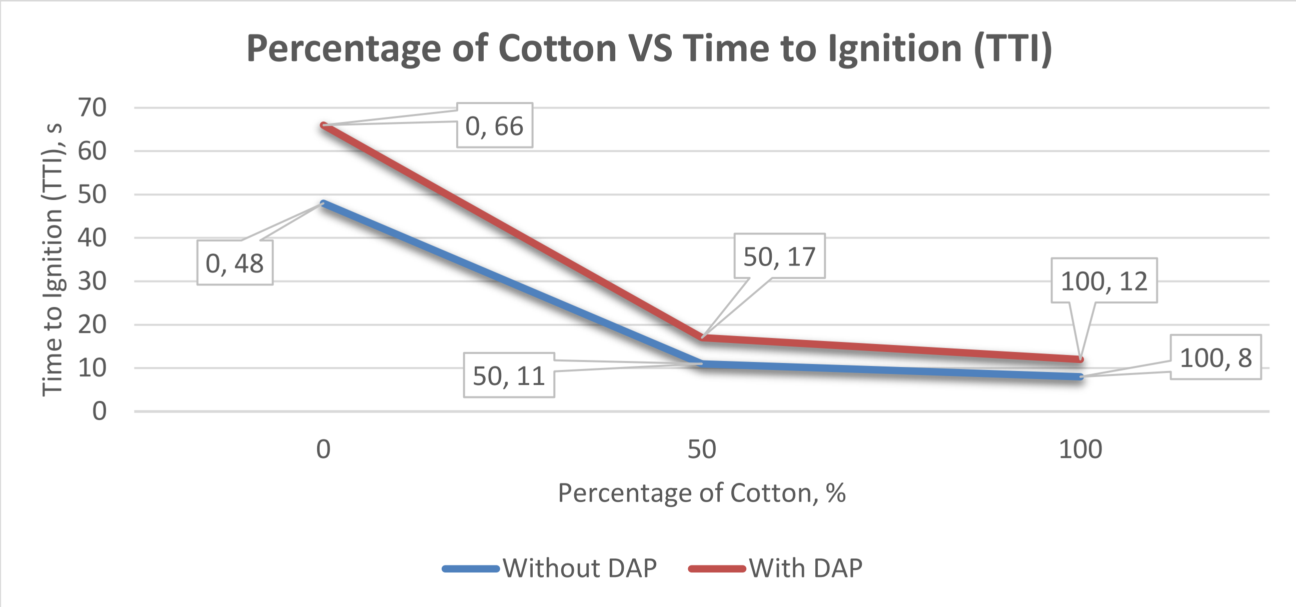


Fig. 1. Result for comparison time to ignition (TTI) with DAP and without DAP

It's worth noting that the absence of cotton in the 0% cotton fabric resulted in the formation of minor chains, which contributed to the anticipation of fabric ignition. This time delay also directly influenced the peak heat release rate (PHRR) and its correlation with the observed durations of sustained combustion as mentioned by White *et al.,* [13]. Consequently, the 100% cotton fabric exhibited a rapid ignition time of 8 seconds, consistent with findings from previous studies by Qi *et al.,* [9]. In contrast, under similar conditions, 50% cotton fabrics showed minimal changes in TTI and PHRR values upon ignition. The remarkable char-forming capacity of the 0% cotton fabric played a crucial role in inhibiting complete combustion by restricting the interaction of combustible materials with lower carbon dioxide levels Qi *et al.,* [9]. These results collectively highlight the effective flame-retardant properties of the printed fabrics in mitigating the propagation of flames.

Many researchers have emphasized the critical role of the peak heat release rate (PHRR) in influencing the onset of flashover in real fire scenarios, especially concerning items like furniture as mentioned Price *et al.,* [7]. The PHRR curves presented in Figure 2 clearly illustrate a non-linear peak that varies in response to the percentage of cotton fabric in the samples.

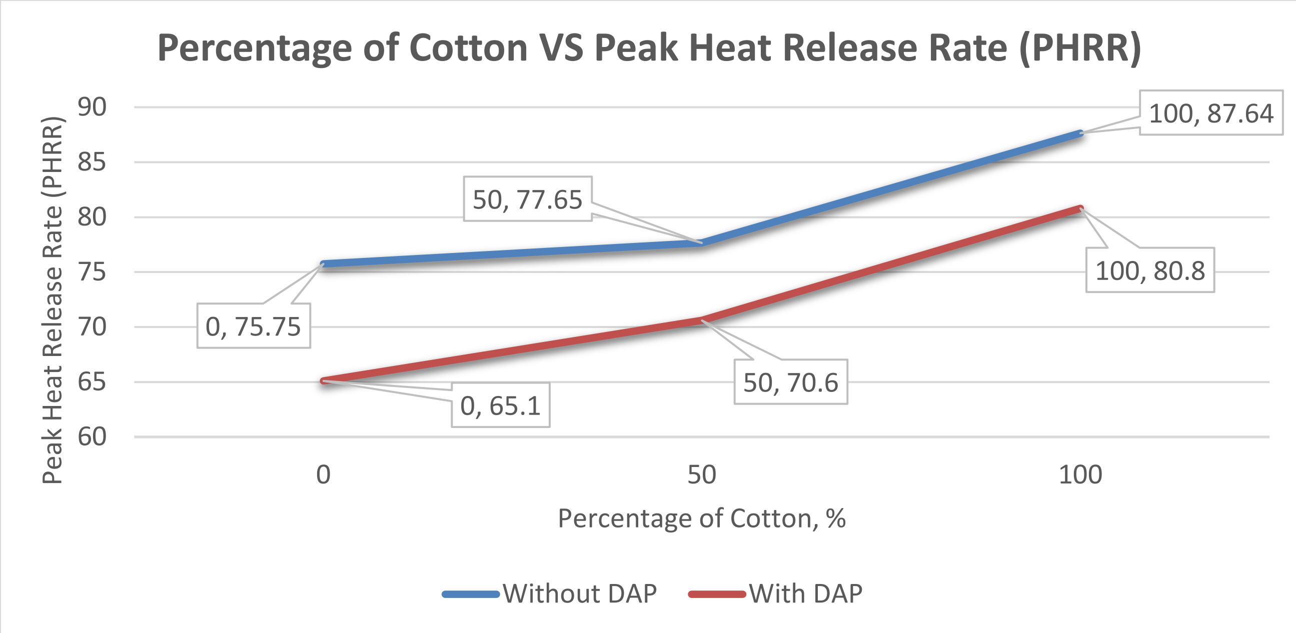


Fig. 2. Result for comparison peak heat release rate (PHRR) with DAP and without DAP

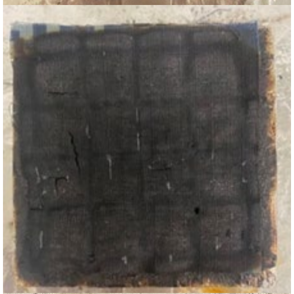
The subsequent increase in PHRR can be attributed to the combustion of the unburned substrate, which is triggered by the fracturing of the initially formed char layer (visually observed). This, in conjunction with an increase in the overall fabric temperature, contributes to the surge in PHRR. Interestingly, a fabric consisting of 100% cotton blended with polyester showed a noticeable reduction in PHRR compared to fabrics composed solely of cotton as proved by Yahaya *et al.,* [11].

Although the tested samples did not exhibit significant differences in flammability under the given heat flux conditions, a discernible trend emerged across the spectrum from 0% to 100% cotton, resulting in a slight increase in PHRR for all fabric samples, both with and without DAP. This trend suggests that the percentage of cotton in the fabric composition may have a subtle influence on PHRR, even though not to a significant degree under these specific testing conditions.

Table 2 provided valuable insights into the test outcomes. It revealed that sample 1 without DAP, which was composed of 100% cotton fabric, was entirely consumed by the fire. While sample 1 with DAP still left behind residue. In contrast, both sample 2 (containing 50% cotton) and sample 3 (0% cotton) that dipped with DAP and without DAP were left behind residues after combustion, forming intumescent char layers. These residues exhibited notable ruptures, indicating that the char layers lacked compactness and mechanical stability, a characteristic discussed in Cheng *et al.,* [3].

Furthermore, a direct correlation was observed is when higher char residue was generated, there was a reduction in the release of flammable volatile compounds. This reduction had the effect of inhibiting the combustion of the fabric, which aligns with findings from previous studies by Price *et al.,* [7]. During the testing process, the samples experienced rapid ignition followed by the determination of a peak heat release rate (PHRR) value. The average heat release rate, on the other hand, represented the fabric's mean level of heat emission during combustion. A larger average heat release rate was indicative of a more intense burning of the material

Table 2
Comparison of Sample Residue after Cone Calorimeter test

Sample	With DAP	Without DAP
Sample 1 (100 % Cotton)		
Sample 2 (50 % Cotton)		
Sample 3 (0 % Cotton)		

3.2 Limited Oxygen Index

Figure 3 in the study presents the Limited Oxygen Index (LOI) results for various fabric compositions, and the data reveals a clear trend where LOI values decrease as the percentage of cotton in the fabric increases. Specifically, the fabric with 0% cotton content had the highest LOI value at 20.6%, while the fabric with 100% cotton content exhibited the lowest LOI value at 17.6%. This trend underscores the fact that polyester is less susceptible to ignition compared to cotton, as supported by earlier research by Yang *et al.,* [15]. In comparison, the 100% cotton that treated with DAP gives better flammability resistance compared to the 100% cotton without DAP since the LOI value is much higher that proved by Khatton *et al.,* [5] and Younis [14].

Yang *et al.,* [15] also found that cotton had an LOI value of 18.4%, indicating its relatively higher flammability, while polyesters had an LOI value of 20.6%, signifying their reduced flammability. This discrepancy can be attributed to the inherent properties of cellulose fibers present in cotton, which are highly prone to ignition as stated by Soliman *et al.,* [12]. Without flame retardants, cotton can readily support the propagation of flames. Textiles made of cellulose fibers, similar to cellulose-based paper, are known to burn easily. In contrast, polyester tends to melt rather than ignite when exposed to flames, moving away from the heat source. In cases where polyester does ignite, the burn is slower than that of cotton and often self-extinguishes. Consequently, burns caused by polyester tend to be deeper but more localized in comparison to cotton. This is importance of considering fabric composition when assessing flammability properties and understanding of the role of different fibers in determining a fabric's susceptibility to ignition and its behaviour during a fire.

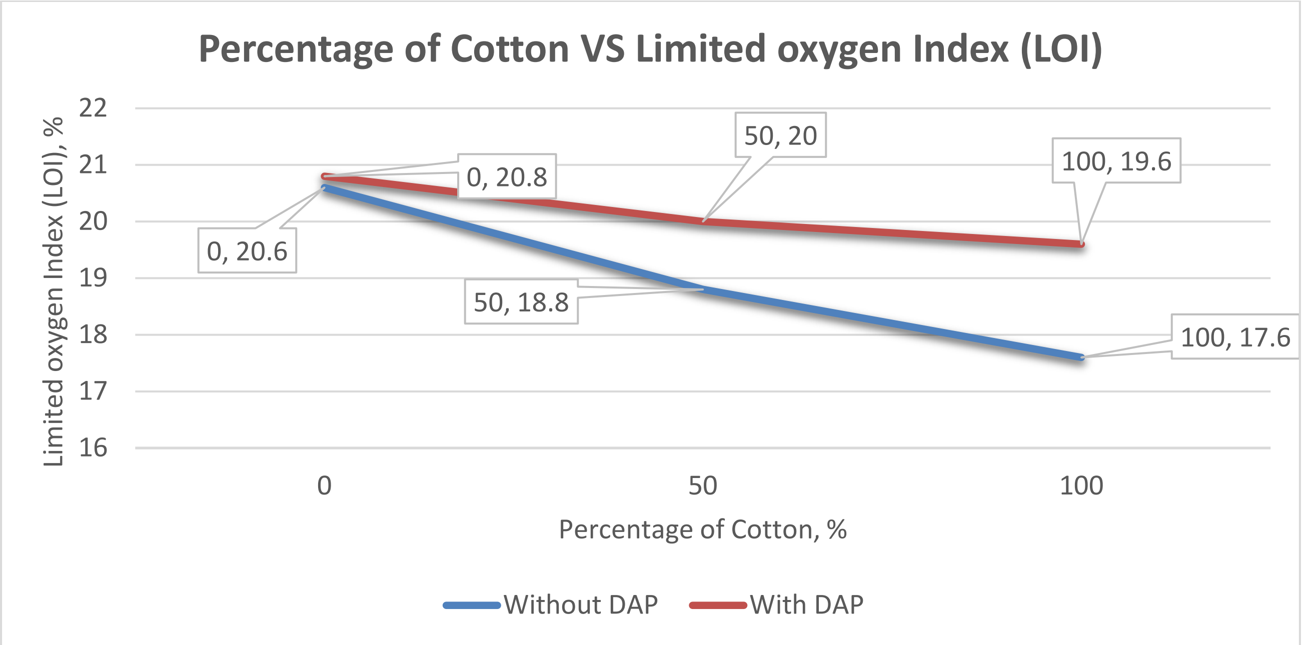


Fig. 3. Result for Limited Oxygen Index (LOI)

4. Conclusions

In conclusion, the findings of this study suggest that an increase in the percentage of cotton in polyester-cotton fabrics is associated with a decrease in flammability properties. This conclusion is supported by the results obtained from the cone calorimeter tests, which demonstrated that fabrics with lower percentages of cotton exhibit better combustibility properties. Additionally, the Limited Oxygen Index (LOI) values indicate that fabrics with higher cotton content are more prone to ignition.

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References

[1] Ahmed, Tamseel, Qamar Mohammed, Balakrishnan Subeshan, Muhammad Rahman, Nurxat Nuraje, and Eylem Asmatulu. "Improving flame retardancy and hydrophobicity of fabrics via graphene inclusion obtained from recycled batteries." *Materials Today: Proceedings* 71 (2022): 78-89. https://doi.org/10.1016/j.matpr.2022.07.469

[2] Razali, Mohd Azahari, Azwan Sapit, Mohd Faizal Mohideen Batcha, Rais Hanizam Madon, Amir Khalid, Md NorrizamMohmad Ja'at, Akmal Nizam Mohammed, Hamidon Salleh, Mohd Faisal Hushim, and UbaidilahAb Hadi. "Preliminary study of effect of natural thread distance on angle dependency of flame spread behaviour over kenaf/polyester fabric." *Journal of Advanced Research in Fluid Mechanics and Thermal Sciences* 62, no. 2 (2019): 250-255.

[3] Cheng, Chen, Su Shuqian, Sun Mingmei, Wang Zhengwen, Zhang Xingrong, and Tang Linsheng. "Synergistic flame retardancy of ZnO with piperazine pyrophosphate/melamine polyphosphate in PP." *Polymer Testing* 117 (2023): 107878. https://doi.org/10.1016/j.polymertesting.2022.107878

[4] Khan, Junaid, and M. Mariatti. "The influence of substrate functionalization for enhancing the interfacial bonding between graphene oxide and nonwoven polyester." *Fibers and Polymers* 22, no. 11 (2021): 3192-3202. https://doi.org/10.1007/s12221-021-1386-y

[5] Khatton, Ayesha, Mubarak Hossen, Nayer Sultana, and Mohammad Lutful Ahsan. "Effect of di-ammonium hydrogen phosphate on jute fabric for fire resistance." *Sci. J. Energy Eng* 7, no. 4 (2019): 63-66. https://doi.org/10.11648/j.sjee.20190704.12
[6] Liu, Yun, Ye-Tang Pan, Xin Wang, Pablo Acuña, Ping Zhu, Udo Wagenknecht, Gert Heinrich, Xiu-Qin Zhang, Rui Wang, and De-Yi Wang. "Effect of phosphorus-containing inorganic–organic hybrid coating on the flammability of cotton fabrics: Synthesis, characterization and flammability." *Chemical Engineering Journal* 294 (2016): 167-175. https://doi.org/10.1016/j.cej.2016.02.080
[7] Price, Dennis, Yan Liu, T. Richard Hull, G. John Milnes, Baljinder K. Kandola, and A. Richard Horrocks. "Burning behaviour of foam/cotton fabric combinations in the cone calorimeter." *Polymer Degradation and Stability* 77, no. 2 (2002): 213-220. https://doi.org/10.1016/S0141-3910(02)00036-8
[8] Suardana, Ngakan Putu Gede, Min Seuck Ku, and Jae Kyoo Lim. "Effects of diammonium phosphate on the flammability and mechanical properties of bio-composites." *Materials & Design* 32, no. 4 (2011): 1990-1999. https://doi.org/10.1016/j.matdes.2010.11.069
[9] Qi, Peng, Yuchun Li, Yuan Yao, Jun Sun, Lu Li, Jian Liu, Xiaoyu Gu, Hongfei Li, and Sheng Zhang. "Ultra washing durable flame retardant coating for cotton fabric by the covalent bonding and interface polymerization." *Chemical Engineering Journal* 452 (2023): 139453. https://doi.org/10.1016/j.cej.2022.139453
[10] Skorodumova, Olga, Olena Tarakhno, Olena Chebotaryova, Oleg Bezuglov, and Fatih Mehmet Emen. "The use of sol-gel method for obtaining fire-resistant elastic coatings on cotton fabrics." In *Materials Science Forum*, vol. 1038, pp. 468-479. Trans Tech Publications Ltd, 2021. https://doi.org/10.4028/www.scientific.net/MSF.1038.468
[11] Yahaya, R., N. Zahari, and WAW Wan Adnan. "Flammability analysis of military fabrics." *Journal of Applied Research in Technology & Engineering* 3, no. 1 (2022): 9-17. https://doi.org/10.4995/jarte.2022.16710
[12] Y Soliman, Marina, and Ahmed G. Hassabo. "Environmentally friendly inorganic materials for anti-flammable cotton fabrics." *Journal of Textiles, Coloration and Polymer Science* 18, no. 2 (2021): 97-110. 10.21608/jtcps.2021.73914.1058
[13] White, Robert H., Sunghyun Nam, and Dharnidhar V. Parikh. "Cone calorimeter evaluation of two flame retardant cotton fabrics." *Fire and Materials* 37, no. 1 (2013): 46-57. https://doi.org/10.1002/fam.2111
[14] Younis, A. A. "Evaluation of the flammability and thermal properties of a new flame retardant coating applied on polyester fabric." *Egyptian Journal of Petroleum* 25, no. 2 (2016): 161-169. https://doi.org/10.1016/j.ejpe.2015.04.001
[15] Yang, Charles Q., Qingliang He, Richard E. Lyon, and Yuan Hu. "Investigation of the flammability of different textile fabrics using micro-scale combustion calorimetry." *Polymer Degradation and Stability* 95, no. 2 (2010): 108-115. https://doi.org/10.1016/j.polymdegradstab.2009.11.047
[16] Haile, Merid, Marcus Leistner, Owais Sarwar, Courtney M. Toler, Robert Henderson, and Jaime C. Grunlan. "A wash-durable polyelectrolyte complex that extinguishes flames on polyester–cotton fabric." *RSC advances* 6, no. 40 (2016): 33998-34004. https://doi.org/10.1039/C6RA03637F
[17] Carosio, Federico, Jenny Alongi, and Giulio Malucelli. "Flammability and combustion properties of ammonium polyphosphate-/poly (acrylic acid)-based layer by layer architectures deposited on cotton, polyester and their blends." *Polymer degradation and stability* 98, no. 9 (2013): 1626-1637. https://doi.org/10.1016/j.polymdegradstab.2013.06.019
[18] Wang, Bin, Ying-Jun Xu, Ping Li, Feng-Qi Zhang, Yun Liu, and Ping Zhu. "Flame-retardant polyester/cotton blend with phosphorus/nitrogen/silicon-containing nano-coating by layer-by-layer assembly." *Applied Surface Science* 509 (2020): 145323. https://doi.org/10.1016/j.apsusc.2020.145323
[19] Carosio, Federico, Jenny Alongi, and Giulio Malucelli. "Layer by layer ammonium polyphosphate-based coatings for flame retardancy of polyester–cotton blends." *Carbohydrate Polymers* 88, no. 4 (2012): 1460-1469. https://doi.org/10.1016/j.carbpol.2012.02.049
[20] Al-Taee, A., Ahlam A. Farhan, and Sadec H. Ghulam. "Effect of flame retardant additive on the flammability of unsaturated polyester resin." *AL-Taqani* 26, no. 2 (2013): 1-8.
[21] Veerappagounder, Sivakkumar, G. Nalankilli, and O. L. Shanmugasundaram. "Study on properties of cotton fabric incorporated with diammonium phosphate flame retardant through cyclodextrin and 1, 2, 3, 4-butane tetracarboxylic acid binding system." *Journal of Industrial Textiles* 45, no. 6 (2016): 1204-1220. https://doi.org/10.1177/1528083714555780
[22] Gaan, Sabyasachi, and Gang Sun. "Effect of phosphorus flame retardants on thermo-oxidative decomposition of cotton." *Polymer Degradation and Stability* 92, no. 6 (2007): 968-974. https://doi.org/10.1016/j.polymdegradstab.2007.03.009