



The flame retardancy of fabricated cotton incorporated with Mg(OH)₂ nano-particles



CrossMark

Alaa S abdelmoaty^{*1}, Ashraf Abou-okeil², Adly A Hanna¹, Ahmed Amr²

¹Inorganic Chemistry Department, National Research Centre, Dokki, Giza, P.O. Box: 12622, Giza, Egypt

²Pretreatment and Finishing of Cellulosic Fabric, National Research Centre, Dokki, Giza, P.O. Box: 12622, Egypt.

Abstract

The aim of this work is evaluation of nano-particles Mg(OH)₂ as flame retardant for fabricated cotton (cotton/PET/50/50). The native samples and that treated with Mg(OH)₂ were characterized by using Infrared (IR) absorption and the Scanning Electron Microscope (SEM). The Mg(OH)₂ nano-particles were incorporated into the fabricated cotton by curing different steps. The appearance of the absorption IR bands at 1443, 1631 cm⁻¹ which specified the Mg-O bands of Mg(OH)₂ unit indicated that Mg(OH)₂ nano-particles are incorporated into the cellulosic chains. On the other hand, the morphology of the studied samples showed that the treated sample with Mg(OH)₂ and the cured temperature not effect on the structure of the native sample. The thermal stabilities of the native and the treated samples were studied. The Thermo-gravimetric (TG) curves showed that a small changes had been appeared by treating cotton/PET fabric with Mg(OH)₂ nano-particles. The curves showed that the treated sample cured at 160°C exhibited less weight loss; this means that this sample is more thermally stable than the other samples. The flammability of the samples was carried out using Limiting Oxygen Index (LOI). The results of the examination show that the value of LOI increases from 20.2 to 23.1 for the sample cured at 160°C. This indicated that Mg(OH)₂ can be used as flame retardant for cotton/PET when cured at 160°C.

Keywords: Flammability; Mg(OH)₂ nano-particles; limiting Oxygen Index (LOI); Thermal stability; Fabricated cotton

1. Introduction

Cotton or grafted cotton fibers with polymer are the most commonly used in textile industries [1-6]. The main drawback in the fiber industry is its flammability, where cotton fibers have low Limited Oxygen Index (LOI equal 18) [7-11]. For these purposes, it is necessary to treat the cotton fibers to resist the rapid flammability. This treatment comes from the grafting cotton fibers with other selected polymers or otherwise treated with flame retardant materials. It noteworthy, that the addition of flame retardant materials to the cotton fibers doesn't effect on the other properties [12].

Different flame retardant materials can be used on the industrial scales. Some organic and inorganic materials can be used for cotton fibers, but the inorganic materials such as Mg(OH)₂, Al(OH)₃, Si(OH)₄ etc [13-16] are more safety than the other materials. Among these materials Mg(OH)₂ powder are used successfully as flame retardant without effects on the mechanical properties like the halogenated and the phosphate materials. In the previous work, the authors studied the effect of the precursor's materials on the morphology and the thermal stability of Mg(OH)₂ to be used as flame retardant materials. They found that the Mg(OH)₂ particles formed in hexagonal structure and have nano-sized particles. The results of the

*Corresponding author e-mail: 3laasalah.86@gmail.com; (Alaa S abdelmoaty).

Receive Date: 16 February 2022; Revise Date: 06 March 2022; Accept Date: 08 March 2022.

DOI: [10.21608/ejchem.2022.122202.5472](https://doi.org/10.21608/ejchem.2022.122202.5472).

©2019 National Information and Documentation Center (NIDOC).

characterization indicate that the more suitable $Mg(OH)_2$ is that samples prepared in presence of surfactants additives [17]. As continuation of this policy, this work aimed to use the $Mg(OH)_2$ nano-particles as flame retardant for fabricated cotton/polyester (PET). The main problem is using $Mg(OH)_2$ as flame retardant for fabricated cotton in the linking of $Mg(OH)_2$ units into the cellulose chains. So that we used special method to attach the $Mg(OH)_2$ molecules.

2. Experimental

2.1. Materials

$Mg(OH)_2$ nano-particles was previously prepared [17]. 50/50% cotton /PET sample were supplied kindly from Misr company, El-Mahala El-kobra, Egypt, 2-propanol, 3-glycidyloxypropyl trimethoxysilane (GPTMS), HCl, Egyptol and 1-methylimidazole were used without purification.

2.2. The Methods of the Experiments

Different steps were followed to attach the $Mg(OH)_2$ nano-particles into the fabricated cotton.

Step 1: $Mg(OH)_2$ powder was dissolved in distilled water by using the ultrasonication route (HP400 S) to obtain homogeneous solution.

Step 2: Preparation of the cross linked agent, hydrolyzed 3- glycidyloxy propyl trimethoxysilane (GPTMS). To complete the hydrolyzed of GPTMS) gel, 20 ml of it was dissolved in 180 ml of 2-propanol followed by adding 180 ml of distilled water (v/v) in glass conical flask with magnetic stirring for 2 hrs at room temperature. During this condition, 2.5 ml of HCl (.01M) were added into the gel solution to complete hydrolyzation.

Step 3: The used samples were washed with 2g/l Na_2CO_3 and 1g/l Egyptol (surfactant)in 100 ml of distilled water suspension and then washed and dried.

Step 4: In this step The cotton fabric/PET sample 50/50 % was treated with $Mg(OH)_2$ in the 100 ml GPTMS to hydrolyzed solution and added 1 to 3 drops 1- methyl imidazol as catalyst then wet pick up of 100% and dried at $100^\circ C/3$ min then cured at $140^\circ C/3$ min. Another sample was cured at $160^\circ C/3$ min as shown in Table 1 and Figure 1.

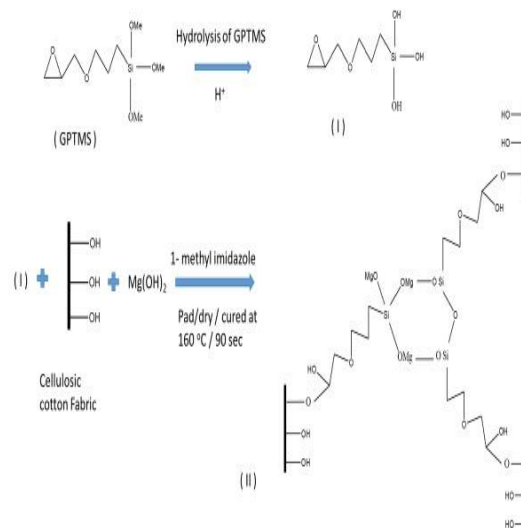


Figure 1. Mechanism of the interaction between Cotton fabric/ $Mg(OH)_2$

2.3.1. Scanning electron microscope

The surface morphologies of the samples were carried out using a Quanta 200 FEG scanning electron microscope (SEM) (Quanta) at an accelerating voltage of 30 kV.

2.3.2. Thermal analysis (TGA)

Thermal analysis (TGA) was performed by USA Berkin – Elmer thermo-gravimeter. Samples of approximately 10 mg were heated from $50^\circ C$ to $800^\circ C$ with heating rate $10^\circ C/min$ under a nitrogen atmosphere, and the flow of nitrogen was 50 ml/min.

Table 1. The Fabricated Cotton Treated with $Mg(OH)_2$ samples curried at two temperatures.

Sample	Cotton fabric (CF)	$Mg(OH)_2/g$	Cured Temperature/ $^\circ C$
Blank	50/50%	0	-
1	50/50%	2	140
2	50/50 %	2	160

2.3.3. Flame retarding test.

The flame retardant properties of the cotton fabrics were tested using limited oxygen index LOI apparatus 4589-2 ASTM D 2863, Elevated-Temperature Oxygen Index ISO 4589-3, UKNaval Engineering standard NES 714 which measure the percentage of oxygen that has to be present the supported burning of the samples.

3. Results and Discussion

3.3. Fourier Transform Infrared (FTIR) Spectroscopy.

Figure 2 represents the IR spectrum of both the fabricated cotton and that treated with $Mg(OH)_2$ as flame retardant. The spectrum in the transmission mode is nearly similar because the bulk composition of the samples is very similar. On the IR profile of the native fabricated cotton (blank), it may specify some peaks characterized the cellulose chain. At 3500 cm^{-1} , it is specified a broad peak characterized the absorbed water on the surface as that attached with the cellulose chain. The band which appears at 3000 cm^{-1} may be specified the stretching of the C-H bond in the cellulose chain. Moreover, it is observed a sharp peak appeared at 1600 cm^{-1} which attributed to the vibration of the carboxylic group ($-COOH$). At 1000 cm^{-1} a sharp peak appeared and attributed to the C-O stretch bonds. The appearance of these peaks specified the cellulosic units [16]. It is noteworthy that these bands still remain after treating with $Mg(OH)_2$ with small changes in the intensity of the peaks. This means that the structure of the cellulosic chain still without any deformation when attached with $Mg(OH)_2$. On the other hand, on the profile of the IR spectrum (Figure 2), we are observed that the appearance of small absorption at $1443, 1631\text{ cm}^{-1}$ which may specified the Mg-O bands of $Mg(OH)_2$ molecule. From this comparison, it may conclude that the $Mg(OH)_2$ molecules are incorporate into the cellulose chain without any deformation in the structure built.

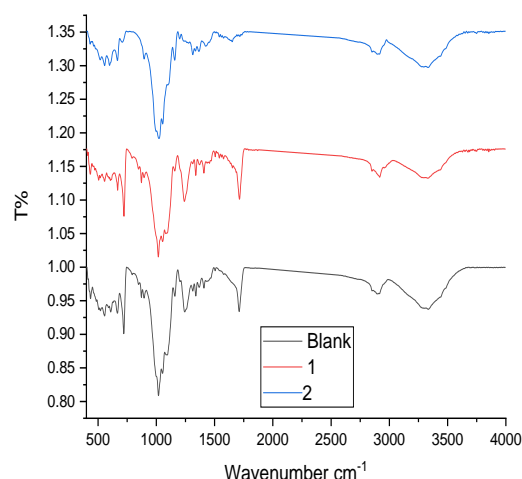


Figure 2. The IR spectrum of both the fabricated cotton and that treated with $Mg(OH)_2$.

3.4. The Thermal Gravimetric Analysis (TGA)

In our previous work [17], it is found that the prepared of $Mg(OH)_2$ nano-particles are thermally stable and can be used as flame retardant materials. In present study, the thermal stability of the fabricated cotton and that treated with $Mg(OH)_2$ are measured. Figure 3 represents the TG curves of the blank sample and other two samples cured at $140\text{ }^\circ\text{C}$ and $160\text{ }^\circ\text{C}$ respectively. It is observed that the thermogravimetric profile behaves the some trends, where it is specified three steps of the thermal weight loss. As reported previously in the literature, these three steps represent the weight loss due to the moisture below $100\text{ }^\circ\text{C}$, the main thermal degradation of the chain between $100\text{-}900\text{ }^\circ\text{C}$ and the last step due to the carbonization of the ash. The TG curves show that the blank cotton fabric and that treated with $Mg(OH)_2$ and carried at $140\text{ }^\circ\text{C}$ are nearly the same, where there is a great difference at $160\text{ }^\circ\text{C}$. These findings indicate that the weight loss for the blank cotton fabric and that cured at $140\text{ }^\circ\text{C}$ are nearly the same. While for the sample cured at $160\text{ }^\circ\text{C}$ at lower in the weight loss was happened as shown in Figure 3. This means that the sample which cured at $160\text{ }^\circ\text{C}$ is more thermally stable [16- 20].

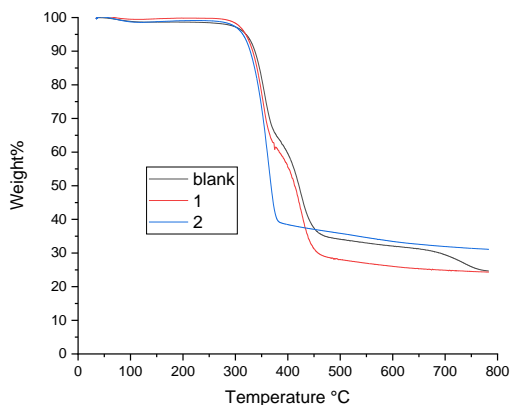


Figure 3. The TGA of both the fabricated 50/50 % cotton and that treated with $Mg(OH)_2$.

3.5. The morphology of the prepared samples.

The morphologies of the Cotton/PET samples and that treated with $Mg(OH)_2$ at two cured temperatures were studied. Figure 4 shows that there is a homogeneous parallel rod arrange for the blank and treated sample. This means that the treated sample with $Mg(OH)_2$ and the cured temperature not effect on the structure of the native sample as conformed from TG curves.

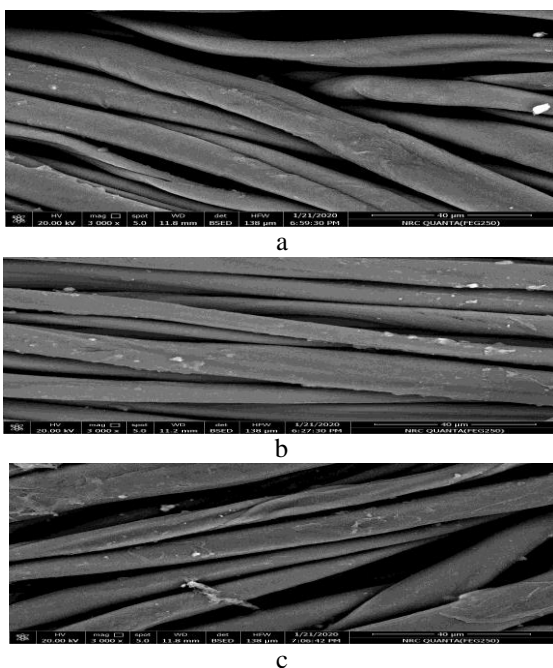


Figure 4. The morphology of treated samples (a) 50/50% Cotton/PET (b) 50/50% Cotton/PET carried at 140 °C (c) 50/50% Cotton/PET carried at 160 °C.

3.6. The flame retardant measurements by Limiting Oxygen Index (LOI).

There are different methods to evaluate the flammability of materials such as UL94, limited oxygen index (LOI), oxygen bomb calorimeter, and cone calorimeter [17, 18]. Among these methods the LOI is acceptable rapid and simple technique. It is well known the air contain 20% of oxygen. The materials fired loss than 20% is flammable materials while that have LOI higher than 20% have less flammable tendency In this work three samples were exposed to LOI for comparison. A blank fabricated cotton/PET and the two samples treated with $Mg(OH)_2$ solution cured at 140 °C and 160 °C. The results of the flammability were recorded in Table 2. The results show that LOI of the blank sample equal to 20.2 where it is higher for the treated samples. For the sample cured at 140 °C the LOI equals 22.6 while that cured at 160 °C equal 23.1. Generally the treatment of cotton fabric samples exhibits higher LOI than 20% which is the percentage in air. This means that the addition of $Mg(OH)_2$ to cotton fabric is effective as flame retardant material.

Table 2. The values of LOI for the samples.

No. of sample	Values of LOI
Blank	20.2
1(cured at 140 °C)	22.6
2(cured at 160 °C)	23.1

4. Conclusion

The analysis of the obtained data by IR absorption and SEM shows that $Mg(OH)_2$ nano particles can be incorporated into the fabricated cotton fibers. Also, it may conclude from the thermal stability data that the samples cured at 160 °C are more stable than the blank sample. The LOI measurements shows that the $Mg(OH)_2$ nano-particles can be used as flame retardant for 50/50 % cotton/PET successfully.

5. Acknowledgment

The authors are thankful to National Research center for continuous advice through this work.

Conflicts of interest

There aren't any conflicts to report.

Formatting of funding sources

The authors acknowledge that financial support was a personal effort.

6. References.

- [1] Yang, M., Liu, W and Liang, A., A mild strategy to construct super hydrophobic cotton with dual self-cleaning and oil–water separation abilities based on TiO₂ and POSS via thiolene click reaction., *Cellulose.*, 27, 2847(2020)., DOI: [10.1007/s10570-019-02963-3](https://doi.org/10.1007/s10570-019-02963-3).
- [2] Mai, Z., Shu, X., Shen, D., and Li, G., One-step fabrication of flexible, durable and fluorine-free super hydrophobic cotton fabrics for efficient oil/water separation., *Cellulose.*, 26,6349(2019)., DOI: [10.1007/s10570-019-02515-9](https://doi.org/10.1007/s10570-019-02515-9).
- [3] Guo, H. and Yang, J., A Robust Cotton Textile-Based Material for High Flux Oil Water Separation., *ACS Applied Materials Interfaces.*,11,13704(2019)., DOI:[10.1021/acsami.9b01108](https://doi.org/10.1021/acsami.9b01108).
- [4] Fan, T., Qian, Q. and Hou, Z., Preparation of smart and reversible wettability cellulose fabrics for oil/water separation using a facile and economical method., *Carbohydrate Polymer.*, 200,63(2018)., DOI:[10.1016/j.carbpol.2018.07.040](https://doi.org/10.1016/j.carbpol.2018.07.040).
- [5] Zhang, M., Wang, C., Wang, S. and Li, J., Fabrication of super hydrophobic cotton textiles for water–oil separation based on drop coating route., *Carbohydrate Polymer.*, 97,59(2013)., DOI: [10.1016/j.carbpol.2012.08.118](https://doi.org/10.1016/j.carbpol.2012.08.118).
- [6] Marzouk, M., Hanna, AA., Abdelmoaty, AS., Abou-okeil, A. and Amr, A., Preparation and characterization of flame retardant cotton fabrics., *Biointerface Research in Applied Chemistry.*,10, 5296 (2020)., DOI: [10.33263/BRIAC102.296300](https://doi.org/10.33263/BRIAC102.296300).
- [7] Mohsin, M., Ahmad, SW., Khatri, A. and Zahid, B., Performance enhancement of fire retardant finish with environment friendly bio cross-linker for cotton., *Journal of Cleaner Production.*, 51, 191(2013)., DOI: [10.1016/j.jclepro.2013.01.031](https://doi.org/10.1016/j.jclepro.2013.01.031).
- [8] Lam, YL., Kan, CW. and Yuen, CW., Objective Measurement of Hand Properties of Plasma Pre-treated Cotton Fabrics Subjected to Flame-Retardant Finishing Catalyzed by Zinc Oxide., *Fiber Polymer.*,15, 1880(2014)., DOI: [10.1007/s12221-014-1880-6](https://doi.org/10.1007/s12221-014-1880-6).
- [9] El-Shafei, A., Elshemy, A. and Abou-Okeil, A., Eco-friendly finishing Agent for cotton fabrics to improve flame retardant and antibacterial properties., *Carbohydrate Polymer.*,118, 83(2014)., DOI: [10.1016/j.carbpol.2014.11](https://doi.org/10.1016/j.carbpol.2014.11).
- [10] Hanna, A.A., Nour, M.A., Elsherief, M.A., Souaya, E.R., Abdelmoaty, A.S, Fire performance of poly propylene treated with ammonium polyphosphate and kaolin., *Egyptian Journal of Chemistry*, 60,5, 937–944(2017)., DOI: [10.21608/EJCHEM.2017.1509.1110](https://doi.org/10.21608/EJCHEM.2017.1509.1110).
- [11] Hanna, A.A., Eglal, R.S., Marwa, A.S., Abdelmoaty, A.S., Studies on the effects of kaolin and modified kaolin on the flammability of APP/PP system., *Egyptian Journal of Chemistry*, 60,2, 205–219 (2017)., DOI: [10.21608/ejchem.2017.593.1005](https://doi.org/10.21608/ejchem.2017.593.1005).
- [12] Łukasz, K., Jolanta, T., Katarzyna, S. and Jeofil, J., Preparation and Characterization of Eco-Friendly Mg(OH)₂/Lignin Hybrid Material and Its Use as a Functional Filler for Poly(Vinyl Chloride)., *Polymers.*, 9, 258(2017)., DOI: [10.3390%2Fpolym9070258](https://doi.org/10.3390%2Fpolym9070258).
- [13] Zheng, D., Zhou, J., Zhong, L., Zhang, F. and Zhang, G., A novel reactive phosphorous flame retardant for cotton fabrics with durable flame retardancy and high whiteness due to self-buffering., *Cellulose.*, 23, 2211(2016)., DOI: [10.1007%2Fs10570-018-1964-3](https://doi.org/10.1007%2Fs10570-018-1964-3).
- [14] Chan, SY., Si, L., Lee, KI., Ng, PF., Chen, L. and Yu, B., A novel boron–nitrogen intumescent flame retardant coating on cotton with improved washing durability., *Cellulose.*, 25, 843(2017)., DOI: [10.1007/s10570-017-1577-2](https://doi.org/10.1007/s10570-017-1577-2).
- [15] Xie, K., Gao, A. and Zhang, Y., Flame retardant finishing of cotton fabric based on synergistic compounds containing boron and nitrogen., *Carbohydrate Polymer.*, 98, 706(2013)., DOI: [10.1016/j.carbpol.2013.06.014](https://doi.org/10.1016/j.carbpol.2013.06.014).
- [16] Abou-Okeil, A., El-Sawy, SM. and Abdel-Mohdy, FA., Flame retardant cotton fabrics treated with organophosphorus polymer., *Carbohydrate Polymer.*, 92, 2293 (2013)., DOI: [10.1016/j.carbpol.2012.12.008](https://doi.org/10.1016/j.carbpol.2012.12.008).
- [17] Hanna, AA., Abdelmoaty, AS. and Sherief, M., Synthesis, Characterization, and Thermal Behavior of Nano-particles of Mg(OH)₂ to Be Used as Flame Retardants., *Journal of Chemistry.*, 2019, 6 (2019)., DOI: [10.1155/2019/1805280](https://doi.org/10.1155/2019/1805280).
- [18] Wanli, J., Hongru, W., Yijun, Y. and Ruirui, W., Mg(OH)₂ and PDMS-coated cotton fabrics for excellent oil/water separation and flame retardancy., *Cellulose.*, 26, 6879 (2019)., DOI: [10.1007/s10570-019-02576-w](https://doi.org/10.1007/s10570-019-02576-w).

[19] Shrivastava, A. and William, A., Introduction to plastics engineering., Cambridge. MA, USA,16,(2018), DOI: [10.1016/B978-0-323-39500-7.00001-0](https://doi.org/10.1016/B978-0-323-39500-7.00001-0)

[20] Mensah, R., Xu, Q., Asante, S., Jin, C. and Bentum, G., Correlation analysis of cone calorimetry and microscale combustion calorimetry experiments., Journal of Thermal Analysis and Calorimetry.,136, 589(2018)., DOI: [10.1007/s10973-018-7661-5](https://doi.org/10.1007/s10973-018-7661-5).