



Activity of inorganic salts on different properties of synthetic leather

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Abstract

As a matter of fact, it has been witnessed that the synthetic leather is becoming the main substitute to its natural leather counterpart. This phenomenon has been observed in the vast majority of fields where these materials are being exploited such as: vehicles interior materials and furniture skin materials. All of which urges the presence of fundamental characteristics that include but not limited to the soft texture, flexibility, mechanical strength and durability. Since these have the responsibility of being easy to burn, flame retardancy is required. In this article inorganic salts with different concentrations are used as flame retardants to synthetic leather as they cheaper and safer than halogenated one; Pot. Persulfate $K_2S_2O_8$ (FRA), Sod. Nitrite $NaNO_2$ (FRB), Di Sod. Tetaborate (Borax) $Na_2B_4O_7$ (FRC), Sod. Sulphite anhydrous Na_2SO_3 (FRD) and Sod. thiosulphite -5-hydroxy $Na_2S_2O_3 \cdot 5H_2O$ (FRE). The current article addresses the focal role of inorganic salts with variable concentrations plays in the process of enhancing the synthetic leather flame retardants. The treated leather was evaluated with different tests, physical properties as thickness, hardness, water vapor permeability and water absorption were determined. In addition, mechanical properties, tensile strength and elongation at break % of the treated leather were evaluated. Thermal stability was determined through, thermal gravimetric analysis (TGA) and the enhancement in the flame retardancy of leather was investigated using horizontal burning test. Correspondingly, delaying firing process help in caring the synthetic leather from burning; led to save money. So the treated synthetic leather be able to use for different manufacturing applications.

Keywords: Flame retardant, synthetic leather, TGA, physical properties, inorganic salts

Introduction:

Synthetic leather is formed by laminating a skin resin layer such as polyurethane, polyolefin, or polyvinyl chloride on a fiber substrate such as woven and non-woven fabric, and knitted fabric. Moreover, an adhesive layer may be interposed between the fiber base material and the skin resin layer. [1-26]

Flame retardant additives used everywhere in marketable products. These materials decrease fire losses by minimizing the probability of ignition and the heat speed release of the products where they are formed. [27-31]

Synthetic leather can be considered as a cornerstone and safe material that is exploited in the majority of luxurious industries such as: upholstered furniture, automotive industry and the footwear industry. This generally always priorities it as number one choice when it comes to health considerations

Synthetic leather used in transportation, cinema and theater seats, car seats, electronics and electrical equipment, furniture and building materials in four areas, every day we are with it "inseparable." [32] The circuit board is coated with flame retardant, mobile phone plastic shell coated with flame retardant using flame retardants, building exterior insulation materials are also coated with flame retardants. Fires happened when a start source, such as a cigarette, stove burner or match meets a flammable fabric such as a polymer, textile, wood leather and papers [33].

The warm from the source breaks down different fabrics, making large endothermically chemical parts and vaporize. At a sufficiently high temperature, these parts react with the oxygen present in the air to release more heat. A few of this warm transmits or changes over back to the fabric, breaking down more, producing more gas- phase fuel, etc. Life- and property- threatening fires result when the rate of

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flame input to the product exceeds the sum of the heat dispersed from the combustion environment leading to pollution of air.

Fire damages to human lives, property and valuables things. From statistics results of fire, it is clear that; there are harms to precious human lives, indicating that products were significantly involved; clothing (including over-suit, undergarment, work wear, suiting etc), upholstered textiles, bed-sheets, floor coverings, leather in seats, and bedding home furnishing were lost.

leather able to be given flame retardant characteristics by introducing elements of inorganic origin. Such elements include sodium, potassium, aluminum, magnesium, and bromine. Inorganic salts flame retardants were added in either the manufacturing process or application through coating or dipping process after manufacturing. Inorganic flame retardants are usually added together or with other types flame retardants to provide a more efficient flame retardant action through synergism.

In recent years, there has been an increasing demand for flame retardancy that does not use halogen-based flame retardants from the viewpoint of environmental protection and the harmfulness of gas generated during combustion. As non-halogen flame retardants, many flame retardants such as ammonium phosphate, ammonium sulfamate, ammonium sulfate, borax, boric acid, aluminum hydroxide, magnesium hydroxide, and phosphate esters are known. [33-35] The integration of flame retardants has the adding of enhancement reaction to fire properties and rising the fire harmful; due to flame spread and heat liberate, may be enlarge the toxicity of the fire emissions or causes other risks due to the chemicals themselves.

Flame retardants can be introduced to many flammable materials to avoid a fire or to delay its start and propagation by interrupting or hindering the combustion process. Thus they save lives, property and the environment. Flame retarded materials often meets the functionality and aesthetic requirements of the consumer as well as offering the most economical approach. The incorporation of fire retardants has the payback of reducing fire reaction and combustion hazards.

Experimental:

Materials:

Synthetic polyurethane leather from leather shop in Kasr- Eleni region, inorganic salts; Pot. Persulfate $K_2S_2O_8$ purity (99 %) and Di Sod. Tetaborate (Borax) $Na_2B_4O_7$ purity (99 %), supplied from Sd. Fine chem. limited Mumbai. Sod. Sulphite anhydrous Na_2SO_3 extra pure (98 %) and Sod. Nitrite $NaNO_2$ purity (98%) supplied from SISCO Research laboratories PVT.LTD, Mumbai, India, Sod. thiosulphite -5-hydroxy $Na_2S_2O_3 \cdot 5H_2O$ BDH purity (99 %) Laboratory supplies Poole, BH15 1TD England.

Methods:

Preparation of different concentrations of flame retardants solutions

Solutions of different salts; potassium persulphate; FRA, sodium nitrite; FRB, di-sodium tetraborate (borax) FRC, sodium sulphite anhydrous FRD and sodium thiosulphate 5-hydrate FRE were prepared by dissolving different concentrations from 0.01 mol to 0.04 mol in 1/2 liter of distilled water from the different inorganic salts. Leather samples with 15 x 20 cm were dipped in for 24 hours. Then leather samples were picked up and let to dry in lab temperature, now it ready to evaluate by different tests. Preparations were shown in Table 1.

Table 1

preparation of solution with different molarities

Molarity, mol	Weight, g				
	potassium persulfate	sodium nitrite	di-sodium tetraborate (borax)	sodium sulphite anhydrous	Sodium thiosulphate 5-hydrate
Abbreviation	FRA	FRB	FRC	FRD	FRE
0.01	1.35	0.345	1.9	0.63	1.24
0.02	2.7	0.69	3.81	1.26	2.48
0.03	4.05	1.035	5.71	1.89	3.72
0.04	5.4	1.38	7.62	2.52	4.96

Characterization

1- Water vapour permeability

Water vapour permeability or (WVP) or is a measure of the passage of water vapor through the material. WVP of synthetic leather treated with different flame retardant solutions was carried out on the Herfeld's appliance (according to DIN 5333 and ASTM-E96) [29]. It is the mass of water vapor absorbed through unit area in unit time under specified conditions of temperature and humidity. ASTM-E96 is one of the most widely used breathability measurement method (the cup/gravimetric method). [36]

Although the standard method suggests range of testing conditions such as temperature, relative humidity and air flow velocity, results reported in practice do not always conform to these suggestions. Herfeld's device consists of a glass container with a metal top cover which contains a circular hole of diameter 60 mm. 50 ml of water is poured into the glass vessel (water method). The studied material pattern is placed (face up) on the cover of the vessel of

circular diameter of 55 mm and the lid is closed and tightened. The device is placed in desiccators with 97 % of sulphuric acid. The weight of the apparatus with the specimen and the water is determined at the beginning and after 48 h. Water vapour permeability (WVP) is determined according to equation:

$$\text{WVP} = [(m_1 + m_2) / 2 - m_0] / tA = G / tA = (G/t) / A$$

G: weight change (g)

m_0 : mass of the device with water and specimen at the beginning (g)

m_1 : mass of the device with water and a test tube after 24 h (g)

m_2 : mass of the device with water and a test tube after 48 h (g)

t: time (h)

G/t: slope of the straight line (g/h)

A: test area (cup mouth area) (m^2)

WVP: rate of water vapor permeability or transmission (g/h m^2) or (mg/h cm^2)

2. Water Absorption (Swelling Character)

Samples were weighed prior to water treatment and then immersed in water for different times. After removal the prepared leather sheets they were wiped with tissue paper to remove excess water on the surface, and then reweighed, the process was repeated after regular time intervals to find out the amount of water absorbed as a function of time. Water absorption capacity (ω) was determined from the relationship.

$$(\omega) = (W - W_0) / W_0 \times 100 = \dots\%$$

Where, W_0 and W are the weights of leather sheet before and after immersion in water respectively.

3. Mechanical Test

Dumbbell-shaped leather specimens were used for the measurement of tensile strength and elongation at break (%) according to ASTM D412c. These tests were carried out using Zwick/Roell (Z10) and cross-head speed of 50 mm/min at room temperature. The test was carried out according to ASTM D412c. Tear strength was measured according to ASTM D624. The average value for each test was taken for three samples to confirm the results (mean \pm SD, $n = 3$).

4. Thermo-Gravimetric Analysis

Thermal analysis (thermal gravimetric analysis [TGA]/DTA) of the leather coated samples was studied with a Perkin Elmer thermogravimetric analyzer, sample size—1–100 mg (typically 5–20 mg), and heating/cooling rate—1–50°C/min (rate = 10°C/min) from room temperature to 600°C at the National Research Center of Egypt.

5. Horizontal Burning Test

Samples with dimension (10 x 100 mm) were exposed to horizontal burning. The flame temperature was ($450^\circ\text{C} \pm 30^\circ\text{C}$), the size of the flame was (30 mm \pm 5 mm) and the diameter of the flame was (5 mm \pm 1mm) for time ranging between 0 till complete burning.

6. Hardness of coated film

The hardness of coated films was measured by Shore Durometer Rubber Portable Handheld Shore A, Digital Shore Durometer A type LCD Display Hardness Meter Tester, range—0–100 HA, resolution—0.5 HA, (TA 300A), China (Zhejiang).

Results and discussion:

Characterization of leather samples

Physical properties:

Thickness and hardness of leather samples (blank and samples treated with different flame retardants) were evaluated and presented in Table (2). From Table (2) it is clear that there are little increases in thickness and hardness by increasing the molarities of solution which didn't affect the quality of samples

Water permeability:

Water vapor permeability of synthetic leather is a combination of the process that water transfers by means of water vapor moves through the hydrophilic groups of inorganic materials. (Table 2) showed that, permeability of the synthetic leather coated by inorganic materials is more hydrophobic than uncoated leather.

The additions of inorganic materials to the synthetic leather samples have an important influence on the water vapour permeability. In the previous table, the water vapor permeability decreased as the molarity of the inorganic substances, which was treated in synthetic leather samples, increased. As the results reported the water vapour permeability were slightly decreased by 3.15% in synthetic leather samples which treated by FR (A, B, C, D and E) the samples with different molarity from 0.01 to 0.04 mol were comparing with the blank sample. The synthetic leather samples which treated by FRB4 and FRC4 and FRE4 were decreased sharply from 0.073 to 0.016 and 0.014, 0.016 respectively. On the other hand, the water vapour permeability as of the synthetic which treated by FRA4 and FRE4 and was decreased to 0.026 and 0.033 respectively.

This decreasing in the water vapour permeability related to the interaction of inorganic flame retardants materials with the surface of the synthetic leather. The physical bond and Van der Waals force between the inorganic materials and PU surface creating a blocking layer in the surface for water transfer channels and reducing the permeability of water vapor of synthetic leather.

From Table 2, it is clear that the thickness and hardness have the same results and the addition have a small effect which doesn't change the final appearance.

Table 2

Thickness, hardness and Water vapor permeability of leather samples

Sample	Thickness, Mm	Shore diameter hardness H A	Water vapour permeability g/h. cm ²
Blank	1.8±0.05	67.6±0.5	0.073±0.003
FRA1	1.81±0.04	68±0.4	0.061 ±0.004
FRA2	1.85±0.03	69±0.5	0.051±0.003
FRA3	1.86±0.02	70.5±0.4	0.042±0.004
FRA4	1.90±0.05	71±0.5	0.026±0.005
FRB1	1.81±0.03	69±0.6	0.067±0.004
FRB2	1.82±0.04	69.3±0.3	0.044±0.003
FRB3	1.84±0.05	70.5±0.7	0.036±0.004
FRB4	1.86±0.03	71.9±0.4	0.016±0.005
FRC1	1.81±0.04	68±0.4	0.038±0.005
FRC2	1.82±0.03	68.6±0.5	0.029±0.003
FRC3	1.83±0.05	68.9±0.3	0.021±0.004
FRC4	1.86±0.03	70.8±0.6	0.014±0.003
FRD1	1.81±0.05	69.5±0.5	0.031±0.004
FRD2	1.81±0.04	70±0.4	0.025±0.003
FRD3	1.81±0.03	71.8±0.6	0.021±0.003
FRD4	1.81±0.02	71.9±0.4	0.016±0.004
FRE1	1.72±0.04	68.8±0.4	0.071±0.006
FRE2	1.72±0.03	69.5±0.5	0.063±0.004
FRE3	1.73±0.05	70.5±0.3	0.053±0.005
FRE4	1.75±0.03	73.5±0.5	0.033±0.004

Where 1= 0.01mol, 2=0.02mol, 3=0.03mol, 4=0.04mol

Water Absorption (Swelling Character):

Samples were weighed prior to water treatment and then wrapped up in water for many times. After taking away the leather samples were wiped with tissue paper to remove excess water on the surface, and then reweighed, the process was repeated after regular time intervals to find out the amount of water absorbed as a function of time. Water absorption ability was determined from the equation.

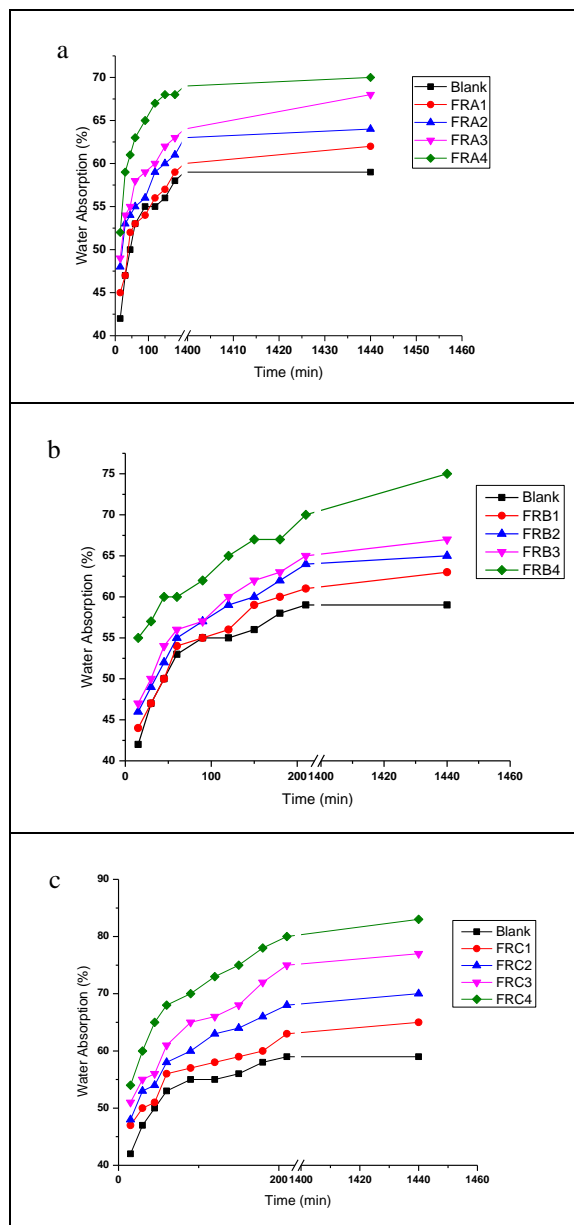
$$\text{Water absorption} = \frac{W - W_0}{W_0} \times 100$$

W_0 and W are the weights of leather samples before and after immersion in water respectively.

All samples show an increase in water absorption by time for blank and treated samples, also water absorption increase with increasing the percentage of morality of salts concentrations.

The water resistance is the most widely calculated parameter of the fiber swell capacity in water. Water absorption in leather samples is directly proportional to the amount of the porosity of leather fibers. The used synthetic leather contains textile layer, which is considered a highly hydrophilic matter. On the other side, the effect of addition of different concentrations of inorganic salts affect the water absorption of treated leather, the results showed in Table (3). it is noticed that all leather sheets samples overloaded with all

inorganic salts have water absorption values higher than the blank which led to decrease in water resistance due to their hydrophobic character of inorganic salts. The results showed that, the water absorption decrease by increase adding of inorganic salts. The samples which treated with borax $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ (FRC) have the highest water absorption one; this may be due to the presence of 10 molecules of water, then leather samples treated was sodium thiosulphite -5-hydroxy $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$; (FRE) and this may be to the presence of 5 molecules in water. Samples which treated with sodium sulphite anhydrous Na_2SO_3 have the less water absorption.



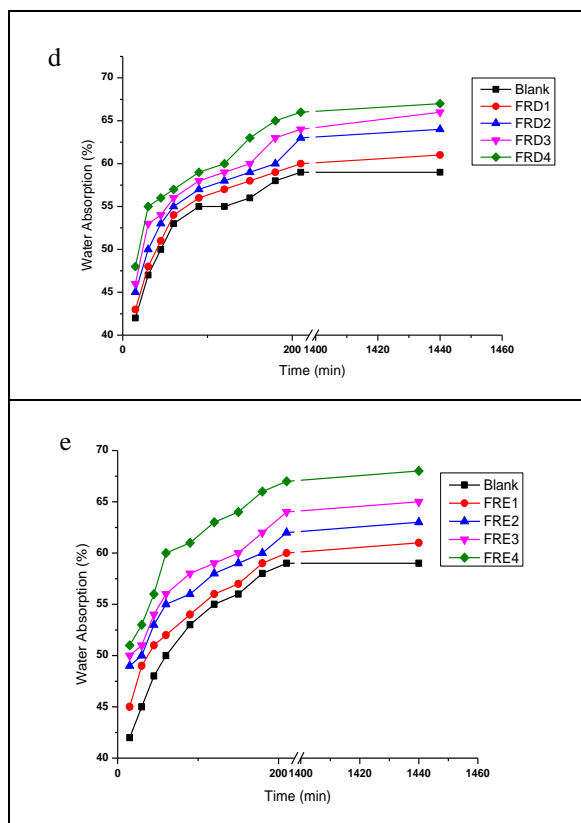


Figure (1) water absorption of synthetic leather coated with (a) FRA, (b) FRB, (c) FRC, (d) FRD and (e) FRE.

2-Mechanical properties

Mechanical properties as well as barrier properties are required for many uses of leather in general, mechanical properties of different leather samples treated with different organic salts have approximately the same effect. The mechanical property frequently measured to characterize leather-samples. Tensile strength and elongation % at break results of treated leather sheets containing different additives concentrations from different materials (FRA, FRB, FRC, FRD, FRE) have higher tensile strength than the blank one expect samples loaded with $K_2S_2O_8$ decrease by increasing the loading concentration while other samples increase with increasing loading percent this may be as a result of the interference of inorganic salts with leather fiber.

Table (4)
Effect of Addition of Inorganic Salts in Tensile strength and Elongation % of leather samples

Sample	Tensile strength Mpa	Elongation at break %
Blank	5.85± 0.33	156± 4
FRA1	5.72±0.32	137.9±3
FRA2	5.66±0.35	142.3±4
FRA3	4.97±0.29	148.66±5
FRA4	4.86±0.27	160.33±6

FRB1	4.93±0.40	153±5
FRB2	5.12±0.39	154±4
FRB3	5.17±0.33	158±5
FRB4	5.24±0.34	163.33±6
FRC1	5.12±0.41	170 ±5
FRC2	5.15±0.37	175 ±5
FRC3	5.44±0.38	177±5
FRC4	5.47±0.36	182 ±6
FRD1	5.41±0.35	169±6
FRD2	5.44±0.38	170±5
FRD3	5.54±0.33	174±6
FRD4	5.61±0.34	175±7
FRE1	5.51±0.34	147.6±3
FRE2	5.76±0.35	146.6±4
FRE3	5.90±0.36	153.16±5
FRE4	6.19±0.34	155.5±5

3- Thermal properties

Thermal Gravimetric Analysis (TGA) of Leather Coated with Inorganic flame retardant materials

TGA analyses of synthetic leather coated with different flame retardants inorganic materials with 0.04 mole ratios FRA, FRB, FRC and FRD were offered in Figure 2 and Table 5. The thermogravimetric curve of synthetic leather is shown in Figure 2(a), from curve there are two-step degradation process, with an $T_{max1}=255^{\circ}C$, $T_{max2}=410^{\circ}C$, in which, the synthetic leather was nearly steady up to $255^{\circ}C$, at which the 1st stage gradual starts to decay involves the decomposition of the textile part adhered to polyurethane (PU) layer. The 2nd stage, about $437^{\circ}C$ is the decomposition of the soft and hard segment of synthetic leather, involves the decomposition of PU leather to PE and diisocyanate. Also, the T_{max50} was reached to $345^{\circ}C$.

Figure 2(b-e) and table 5 shows The TGA curves of synthetic leather with different inorganic flame retardants FRA, FRB, FRC and FRD exhibit also two distinctive weight loss stages, from a range of $220^{\circ}C$ to about $600^{\circ}C$. The 1st stages, in range of $263-275^{\circ}C$ is the primarily decomposition of the textile part adhered to PU. The 2nd stages, about $420^{\circ}C-440^{\circ}C$ related to the decomposition of the soft and hard segment of polyurethane, involves the dissociation of urethane to amines, alkenes and carbon dioxide.

The TGA results presented in Figure 2(b-e) showed that, the degradation composition temperature of the synthetic leather coated with different inorganic flame retardants FR(A-D) is slightly higher than uncoated synthetic leather. This may be due to the incorporation of the inorganic materials into synthetic leather which increases the thermal stability of the PUs leather over that of the uncoated synthetic leather. This slightly enhancement in thermal stability recognized to the physical bond between inorganic materials and PU polymer. This can be explained by brought about multiple weak hydrogen bonding between the

carbonyl groups (C=O) of the PU and the hydrogen atoms of inorganic materials.

The incorporation of different amount of inorganic materials didn't develop sharply the first degradation temperature; however, increase the second degradation temperature. This indicates that inorganic materials cooperate with the soft segment in PUs configuration which restrictions the segmental motions of PU resulting in the rising in the thermal stability. [36-39]

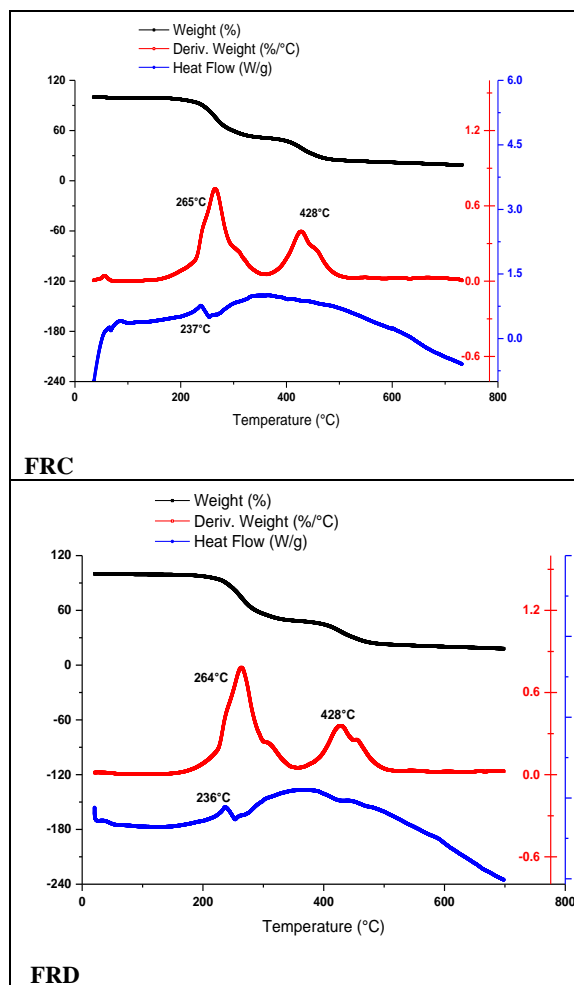
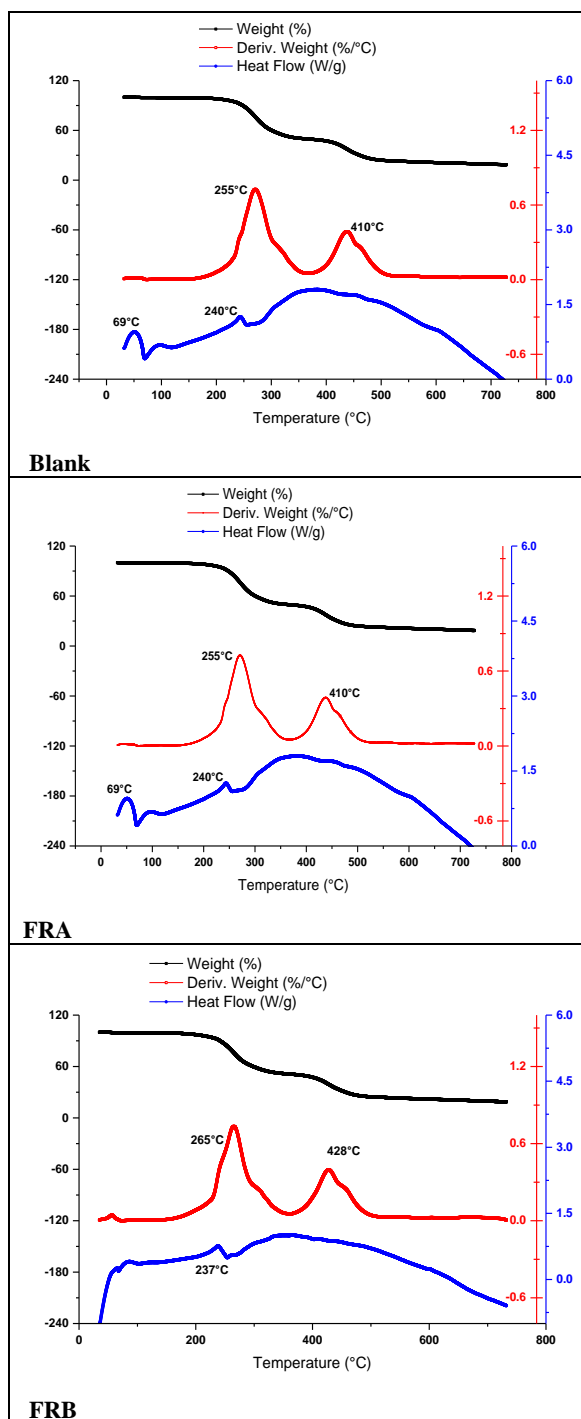


Fig. 2 : TGA of Leather Coated with Inorganic flame retardant materials

Table (5)
TGA of Leather Coated with Inorganic flame retardant materials

Sample	DSC, °C	TGA, °C	T _{max50} , °C
Leather Blank	T _{g1} =62.86 T _{g2} =243	T _{max1} =255 T _{max2} =410	345
Leather+FRA	T _{g2} =240	T _{max1} =263 T _{max2} =426	355
Leather+FRB	T _{g2} =237	T _{max1} =265 T _{max2} =428	375
Leather+ FRC	T _{g2} =244.9	T _{max1} =275 T _{max2} =436	360
Leather+ FRD	T _{g2} =237	T _{max1} =264 T _{max2} =428	360

4- Flammability:

The results of flammability of effective various flame retardants are listed in Table (6). From Table (6) it is can be noted that; all treated samples have flame retardancy more than the blank one, the blank one take

29 sec. to be extinguished while samples treated with $K_2S_2O_8$, (FRA) the time to extinguishment decrease to 6,6 sec.- 5.86 sec.-2.58 and reach to 1.52 sec. at 0.04 mol this may be due to the presence of sulfur which help in enhancement retardancy. Samples treated with Sod. Nitrite $NaNO_2$ (FRB) take 10.5 -1.86 sec. to extinguished then - Self extinguishment by increase concentration of salt; this improve in retardancy may be due to presence of nitrogen.

Samples treated with (FRC) (Borax) $Na_2B_4O_7 \cdot 10 H_2O$ Flame retardancy decrease by increase concentration of salt, 19 sec. - 15.5sec - 9.5sec. Self extinguishment this due to the presence of 10 H_2O which help in extinguishment the flame.

Samples treated with (FRD); Na_2SO_3 ; Also all samples decrease by increasing salt content due to increase in sulfur content from 10 sec., 7 sec., 5 sec, to Self-extinguishment.

Samples treated with (FRE) $Na_2S_2O_3 \cdot 5H_2O$ have the best retardancy due to the presence of both sulfur and 5 H_2O . 0.01 take 14 sec. the all concentration are self extinguishment.

Table (6)

Flammability measurements of effective different flame retardants

Sample	Flammability, sec.
Blank	29
FRA1	6,6
FRA2	5.86
FRA3	2.58
FRA4	1.52
FRB1	10.5
FRB2	1.86
FRB3	Self extinguishment
FRB4	Self extinguishment
FRC1	19
FRC2	15.5
FRC3	9.5
FRC4	Self extinguishment
FRD1	10
FRD2	7
FRD3	5
FRD4	Self extinguishment
FRE1	14
FRE2	Self extinguishment
FRE3	Self extinguishment
FRE4	Self extinguishment

Conclusion

All on all, the study displayed a significant success in treating synthetic leather which have flame retardant properties, this was effectively achieved via the exploitation of relatively inexpensive inorganic salts such as; $K_2S_2O_8$, $NaNO_2$, borax, Na_2SO_3 and $Na_2S_2O_3 \cdot H_2O$ which have the ability to decrease

flammability of treated synthetic leather. After synthetic leather treatment with different concentrations of inorganic salts; samples treated with high dose of Na_2SO_3 and $Na_2S_2O_3 \cdot H_2O$ the leather samples were self-extinguishing, in the same time keeping physical and mechanical properties without deteriorations. In addition, improvements of some properties such as tensile, elongation % and water absorption were also observed as leather samples treated with borax, Na_2SO_3 and $Na_2S_2O_3 \cdot H_2O$, the price of this three inorganic salts are approximately in same rang. Delaying firing process help in protecting the materials from burning, saving human lives, property and valuables thing which reflect in the economic side. So the treated synthetic leather can be used for different industrial applications as safer product. This approach can be also extended to various filed of chemical industry as polymer, rubber and plastic.

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نشاط الأملاح غير العضوية على الخصائص المختلفة للجلد الصناعي

في الواقع ، لقد لوحظ أن الجلد الصناعي أصبح البديل الرئيسي لنظيره من الجلد الطبيعي. وقد لوحظت هذه الظاهرة في الغالبية العظمى من المجالات التي يتم فيها استغلال هذه المواد ، مثل: المواد الداخلية للسيارات والأثاث والمواد الجلدية. كل هذا يحث على امتلاك الخصائص الأساسية التي تشمل على سبيل المثال لا الحصر الملمس الناعم والمرونة والقوة الميكانيكية والمتانة ونظرًا لأن الجلد الصناعي يحتوي على مواد سهلة الاحتراق، فإن استخدام مثبطات اللهب لها أهمية كبيرة ومطلوبة. في هذه المقالة تستخدم الأملاح غير العضوية ذات التركيزات المختلفة كمثبطات للهب للجلد الصناعي لأنها أكثر أمانًا من الأملاح الهالوجينية مثل البوتاسيوم بيرسلفات (FRA)، نترات الصوديوم (FRB)، البوراكس (FRC) ، صوديوم سلفيت (FRD) و هيدروكسي صوديوم ثيوكيريتيت (FRE) . تم تقييم الجلد المعالج باختبارات مختلفة ، وتم تحديد السماكة والصلابة ونفاذية بخار الماء وامتصاص الماء. بالإضافة إلى ذلك ، تم تقييم الخواص الميكانيكية وقوة الشد والاستطالة % للجلد المعالج . تم تحديد الثبات الحراري من خلال التحليل الوزني الحراري (TGA) وتم فحص تعزيز مثبطات اللهب للجلد باستخدام اختبار الاحتراق الأفقي. في المقابل ، يساعد تأخير عملية الاحتراق في العناية بالجلد الصناعي من الحرق أدى إلى إنقاذ العديد من الاموال. لذلك يمكن استخدام الجلد الصناعي المعالج في تطبيقات التصنيع المختلفة.