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Fire Retardant Carton by Adding Modified Leather Waste

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ONE of the greatest considerable problems of the leather industry is waste generation. About 30% of leather substance processed in tanneries is rejected, mainly after shaving process, in the form of protein waste containing about 10–15% chromium (III). This waste is generally deposited and burned causing hazards to all environment; man, plant and animal. In this work; this waste will be grinded to nanosize, treated with flame retardants then added as filler during the formation of carton sheets. The prepared carton sheets were evaluated using scanning electron microscope (SEM) and thermal gravimetric analysis (TGA). Furthermore, the physical properties as basis weight, sheet thickness, airermeability, density of paper sheet were evaluated. The optical properties of the prepared carton sheets were examined. In addition, the mechanical properties (breaking length, tear factor and burst factor) of the prepared carton sheet were assessed. Moreover, the carton sheets were examined as fire retardants materials. Therefore, these carton sheets can be used as materials for packaging applications.

Keywords; Flame retardant, Unbleached rice straw, Carton sheet, Packaging, SEM and TGA.

Today, carton and coated paper with polymer has great consideration in novel functional carton and papers for several application such as packaging, for example anti-static and electro-magnetic shielding papers, new wall coverings, and antibacterial papers [(1-3)] .

The development of science and technology offers the obtainability of sophisticated product but currently increase the use of combustible materials. Paper and carton are commonly used in everyday life and this increase fire hazard, the need to treat with flame retardant to limit their ability firing [(4-5)].

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Various methods can be used to protect materials more effectively against attack by fire, first the materials those inherently containing P, Si, B, N or others contain miscellaneous element; itself is fire retardant as inorganic material. Second; incorporation of flame retardant additives via copolymerization or some type of chemical modified, *e.g.* grafting or by physical crosslinking. This can cause changes in physical and mechanical properties of the material(6). Third type is material containing flame retardant as exhibit an endothermic decomposition cooling and diluting the ignitable gas mixture owing to formation of inert gases accompanying with the formation of an inert gases associated with the formation of an oxide protective barrier(7-9). Moreover, fibrous raw materials for pulp making are divided into three main categories ; wood fibers that constitute about 75% of all fibrous raw supply of pulp mills reclaimed waste paper about 20% and remaining 5% for the increasing demands coupled with increasing shortage and higher prices of wood, nonwooding fibers gained. Their importance as raw material for pulp production in countries in wood sources (10-15) .

Correspondingly, leather industry generate large amounts of waste, most of them are burned causing environmental pollution, in the last few years, studies were directed to utilize these waste as fillers in rubber(15), or extracted gelatin from them(14). In previous studies these waste were after grinding and treated with different agents and used as filler in paper making aiming to enhance their mechanical, physical and fire retardancy properties(17-20). Furthermore, many natural or synthetic types of filler can be replace paper pulp aiming to preparing some specialty carton papers or for preparing carton papers with less expensive cost. Every 1% increase in fillers decrease the cost by 2.5$/ton (21).

Our goal is inhibiting or even reducing the flame retardancy through incorporation of nanoparticles of leather waste treated with different flame retardant material during carton sheet formation. The results show that; leather waste nanoparticles treated with flame retardant material exhibit superior flammability inhibiting properties without destroying the mechanical properties of carton sheets.

Experimental

Raw material

The leather waste were supplied from medium tannery in Misr Alkadima Region. The bagasse pulp was supplied by Edfo Company. The triethyl phosphate (TEP) were purchased from Gmbh (Germany).

1 . Preparation of the treated leather powder

Leather waste shavings were disintegrated in a multistage way to obtain a powder then sieved through 0.3 meshes as in previous work(16). The resultant leather powder was divided into three parts: untreated leather powder, leather powder treated with either triethyl phosphate (treated I) and Bromin (treated II). The treated samples were filtered and dried at 60˚C for 2 hr in an air oven and sieved again through 0.3 meshes. They were then included in the preparation of carton sheets.

2 . Preparation of the pulp

Pulp samples were impregnated in water for 24 hr, and then beated in a Valley beater, the beating process was carried out at 10% pulp consistency at a speed of 150 r.p.m. and beaten to 35 °SR using a Valley beater. After beating, the pulp was transferred to a 2 liter measuring cylinder. The stock dilutes with water to 2000 ml and processed in a disintegrator for 2 min at 3000 r.p.m. the carton paper sheets was prepared in lab.

3. Hand sheet preparation and characterization

Different amounts (6, 9, and 12 %) of leather waste, treated and untreated, were added to bagasse pulp suspension at 4 % consistency and stirred for 30 min. The mixtures were then diluted to 2% consistency and stirred at about 1000 rpm for 30 min to ensure homogenous distribution of leather waste. Carton sheets (mass = 2 g) were made using laboratory sheet former according to the scan standard method (scan 1976). After forming, the sheets were pressed for 5 min at 420 kPa at 80°C. Ten conventional hand sheets with a basis weight of about 180 $g.m^{-2}$ were prepared on a Rapid Khöten sheet former, following the ISO 5269-2 standard method. Regarding their physical properties, the hand sheets were conditioned at 23°C and 50 % of relative humidity before testing, as recommended by the ISO 187 standard. Then, the basis weight (ISO 536), thickness (ISO 534), bulk and permeability (ISO 5636-3) were measured. Finally, the main mechanical properties were assessed according to ISO standard methods. The burst factor was measured on Lhomargy equipment. The tear factor was evaluated on anLhomargy ED20. The optical properties were measured with a Color touch spectrophotometer (Model isoTechnidyne corporation, New Albany, Indiana, USA). The air permeability was carried out on a Lorentzen and Wettre equipment. Others blank carton paper sheets were prepared using a hand sheet former from neat bagasse fiber beaten to 45 and 35^{o}SR, respectively using valley beater.

Characterizations of carton paper sheets

1. Physical properties

The basis weight (ISO 536), thickness (ISO 534) and permeability (ISO 5636-3) for paper sheets were measured. Film thickness was measured with a 1 µm precision with a hand-held digimatic micrometer (Quantu Mike Mitutoyo). Four thickness measurements at different positions were taken on each specimen. The airermeability was carried out on a Lorentzen and Wettre equipment. Density of paper sheet determined according equation D=W/volume. The optical properties were measured with a Color touch spectrophotometer (Modelisotechnidyne corporation, New Albany, Indiana, USA).

2. Mechanical testing

The main mechanical properties were assessed according to ISO1924-3 standard methods; tensile tests for the paper sheets were carried out on a RSA3 (TA Instruments, USA) equipment working in tensile mode. The measurements were accomplished at room temperature (~25°C), with a distance between jaws of 10 mm, cross head speed of 0.6 $mm.min^{-1}$ for the first 250 s, then 1.5 $mm.min^{-1}$ up to 2000 s, and finally 3 $mm.min^{-1}$ up to the break; five replicates were tested for each film. The sample dimensions were 20 mm long and 5 mm wide.

3. Flame test: Home developed method

The test device is a UL 94 flame chamber. The sample dimensions were 150x50 mm^2. The flame height was 20 mm. The samples were located at an angle 45^o. The flame was applied for 2 s at 19 mm from the bottom edge. Burning time and length were recorded.

4. Scanning electron microscope

The samples ($1cm^2$) were subjected to sputter coating of gold ions which act as conducting medium during scanning with Jeol scanning microscope (type JXA-840A, Japan) with accelerating 20.00kV.

5. Thermo gravimetric analysis

The thermal properties of treated and untreated samples were carried out using a TGA Perkin Elmer, with a rate of 10°C/min. The temperature ranged from room temperature up to 500°C under nitrogen atmosphere.

Results and Discussion

The results of basis weight, thickness , the mechanical properties; (tear, burst, breaking length young modules and elongation%) also the physical properties; (brightness ,opacity and air preamiabilty) of the prepared carton sheets, issued from bagasse as cellulosic raw materials and that loaded with different concentrations of untreated leather waste, are presented in Table 1.

The mechanical properties of the prepared carton sheets; the tensile, young modules and elongation%, the tensile of the carton sheet which prepared using untreated leather start to increase by rising the amount of untreated leather (6 % , 9% and 12 %) this is due to presence of leather nanoparticles that intercalated between the cellulosic fibers which enhance the mechanical properties of the prepared carton sheet. Also; the young modules and elongation enhanced by the addition of untreated leather is shown in Table 1. The physical properties for example (brightness , opacity and air permeability) rising by increasing the percentage of addition leather waste. The opacity have not significant change from the blank through the different concentration of the untreated leather (6, 9 and 12%), while the brightness of the prepared paper sheet increase by the increase of the concentration of untreated leather. There is an obvious increase in air permeability; due to presence of leather nanoparticles as filler between cellulosic fiber help in presence interspaces so air permeability increase.

TABLE 1. Physical properties of blank carton sheet as well as the prepared carton sheet containing different concentrations of unmodified leather, 6, 9 and 12 %.

Type of Tests	Blank	carton sheet with 6%UML	Carton sheet with 9% UML	carton sheet with 12% UML
Basis weight (g/m^2)	195.55± 4.56	201± 3.97	201.16± 5.4	200.7± 5.09
Tear ($mN.m^2/g$)	4.24± 0.59	4.64± 0.055	4.70± 0.12	4.75± 0.17
Burst ($kPa.m^2/g$)	3.40± 0.22	3.44± 0.44	3.48± 1.49	3.98± 0.26
Thickness (µm)	240± 7.83	238.80± 5.87	281± 28.88	261.8± 8.88
Air permeability ($ml/S.cm^2.Pa$)	0.123± 0.015	0.198± 0.027	0.242± 0.029	0.254± 0.014
Opacity (%)	99.41± 0.16	99.31± 1.03	99.62± 0.199	99.2± 0.88
Brightness	43.61± 0.01	44.87± 0.65	45.24± 1.1	44.35± 2.40
Breaking length (kg/cm^2)	4.35± 3.60	8.63 ±5.37	6.71 ±5.20	9.50 ±5.20
Elongation (mm)	13.16±1.87	14.70 ±2.67	12.12 ±2.39	17.36 ±2.47
E-modules (GPa)	6.20±3.61	5.44± 7.93	4.66± 4.1	4.94± 5.5

Along with treated leather waste (TEP and Bromine treatments) are revealed in Tables 2 and 3, it can be noted that, the burst factor didn't have obvious change while breaking length increase steadily by increasing the concentration of treated leather waste. Correspondingly, the tear factor increases by the concentration addition of treated as shown in Tables 2 and 3; this enhancement in tear for carton sheet may be due to intercalation between the cellulosic fibers and leather waste fiber which reflect on its strength. Furthermore, the brightness of the prepared carton sheet using treated leather; there is slight increase in case of leather treated with bromine with 9% and 12% concentrations owing to the white color of bromine. While the opacity of carton sheets increased with increasing the loadings % of treated and untreated leather. The results for air permeability are presented in Tables 2 and 3. The air permeability improved gradually by increasing loadings of the addition for both treated leathers. This increase in air permeability is due to the occurrence of interspecies between leather fiber and cellulosic fiber.

TABLE 2. Physical properties of blank carton sheet as well as the prepared carton sheet containing different concentrations of first modified leather (MLI), 6, 9, and 12 %.

Type of Tests	Blank	carton sheet with 6%MLI	carton sheet with 9% MLI	carton sheet with 12% MLI
Basis weight (g/m^2)	195.55± 4.56	198.4± 3.84	193.97± 2.63	199.37± 2.54
Tear ($mN.m^2/g$)	4.24± 0.59	4.71± 0.06	4.75± 0.042	4.79± 0.11
Burst ($kPa.m^2/g$)	03.40± 0.22	3.48± 0.21	3.89± 0.27	3.93± 0.18
Thickness (μm)	240.00± 7.83	259.8± 4.1	261.8± 3.29	273.4± 5.10
Air permeability ($ml/S.cm^2.Pa$)	0.123± 0.015	0.243± 0.013	0.293± 0.013	0.28±0. 89
Opacity (%)	99.41± 0.16	97.88± 0.14	99.73± 0.12	99.69± 0.25
Brightness	43.61± 0.01	44.79± 0.91	44.68± 0.72	44.99± 1.08
Breaking length (kg/cm^2)	4.35± 3.6	5.61± 0.48	4.5± 0.47	4.77± 0.48
Elongation (mm)	13.16 ± 1.87	12.61± 0.09	12.46± 0.46	12.68± 0.69
E-modules (GPa)	6.2±3.61	5.23± 0.36	4.78± 0.25	4.3± 0.49

TABLE 3. Physical properties of blank paper sheet as well as the prepared paper sheet containing different concentrations of second modified leather (MLII), 6, 9, and 12 %.

Type of Tests	Blank	Paper sheet with 6%MLII	Paper sheet with 9% MLII	Paper sheet with 12% MLII
Basis weight(g/m^2)	195.55± 4.56	200.28± 4.22	203.38± 3.54	199.37± 2.54
Tear ($mN.m^2/g$)	4.24± 0.59	4.80± 0.145	4.76± 0.10	4.71± 0.69
Burst ($kPa.m^2/g$)	3.40± 0.22	3.47± 0.16	3.90± 0.178	4.48± 1.11
Thickness (μm)	240± 7.83	259.8± 4.1	261.8± 3.29	273.4± 5.10
Air permeability ($ml/S.cm^2.Pa$)	0.123± 0.015	0.218± 0.18	0.212± 0.098	0.25± 0.168
Opacity (%)	99.41± 0.16	99.11± 0.112	98.96± 0.86	98.91± 0.17
Brightness	43.61± 0.01	45.83± 0.733	46.69± 0.425	46.65± 0.504
Breaking length (Km)	4.35± 3.6	5.27±8.85	5.41± 7.72	5.8± 0.48
Elongation (mm)	13.16± 1.87	14.92 ±2.16	19.13 ±2.6	21.12 ± 1.45
E-modules (GPa)	6.2 ±3.61	5.38 ±5.19	5.33 ±4.26	4.77± 0.269

Scanning electron microscope

The structure of the prepared carton sheets were investigated in details by using scanning electron microscope (SEM). The scanning electron microscope for carton sheets were carried out using fractural and surface images for blank carton sheet as well as the treated carton sheet and the image represented in Fig. 1(a-c). From the Fig. 1a, the blank sheet apparent some porous areas and fractural image reveals the homogeny of the fibers inside the carton sheet also the surface image showed that the surface completely smooth and homogenous. However, the Fig. 1b displayed the carton sheet prepared by using different concentrations of unmodified leather, the appeared pore in blank carton sheet was filled by unmodified leather as shown in both fractural and surface image and the filling increased with the concentration of unmodified leather increased. The untreated leather existing on both the cellulose fibers and the surface pores of carton may be responsible for the increase of the thermal stability and enhance the fire resistance.

The same observation for treated leather (treated I and treated II), the treated leather by nanomaterials distributed not only on the surface of the carton sheet but also between the fibers of the prepared carton sheet and the filled the pores in carton sheet. The filling rising by increasing the concentration of treated leather from 6 to 12 %. Fig. 1(b and c) displayed the morphology of the carton sheets containing different ratios of treated leather with different nanomaterials. The images show the modification of the surface of carton sheet which become smoother due to the leather blockage the pores. Furthermore, by using the nanomaterials treated leather with different concentrations, the SEM images Fig.1 (b and c) demonstrated the incorporation of the filler particles between the carton sheet pores which could be the enhancement of carton sheet flame retardant owing to this reason.

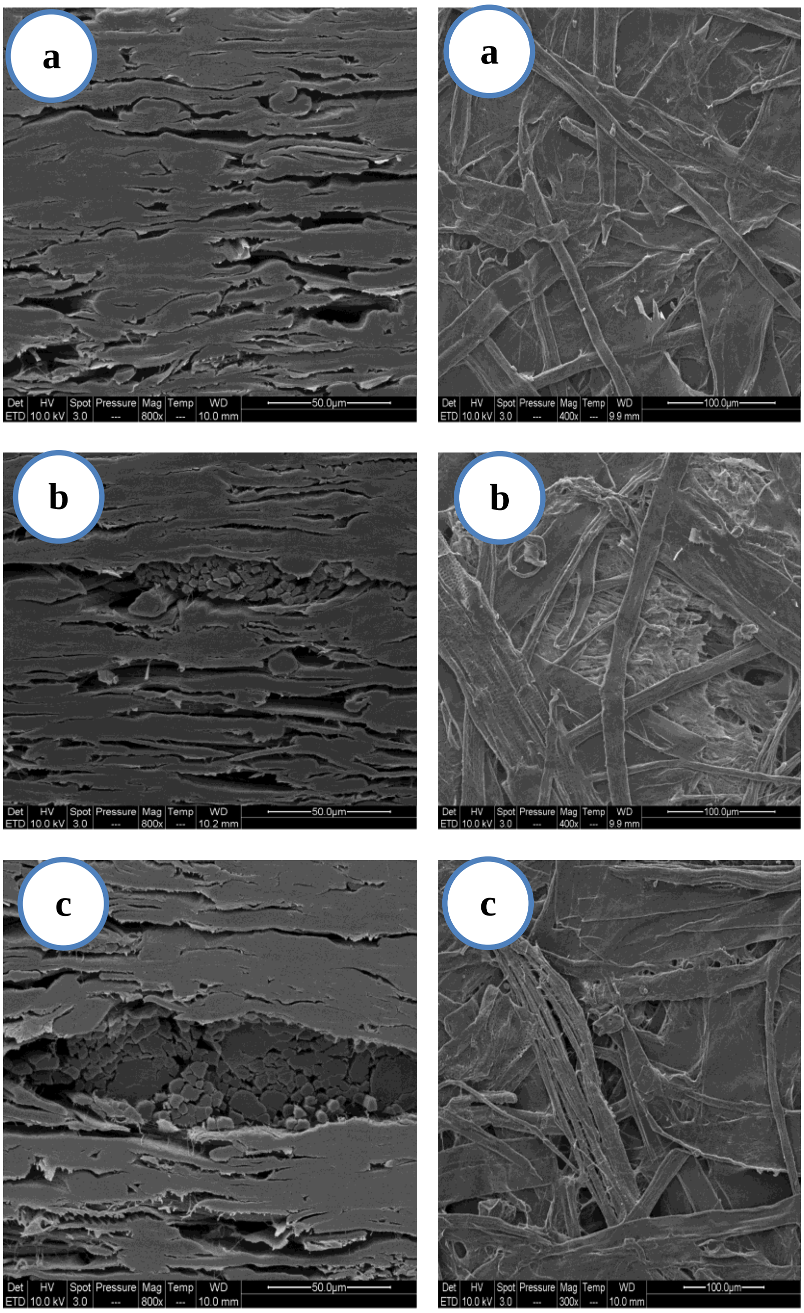

Fig. 1a. SEM images of a) blank carton sheet with fracture and surface image as well as carton sheet with different ratios of unmodified leather, b) 6% and c) 12% with fractural and surface image.

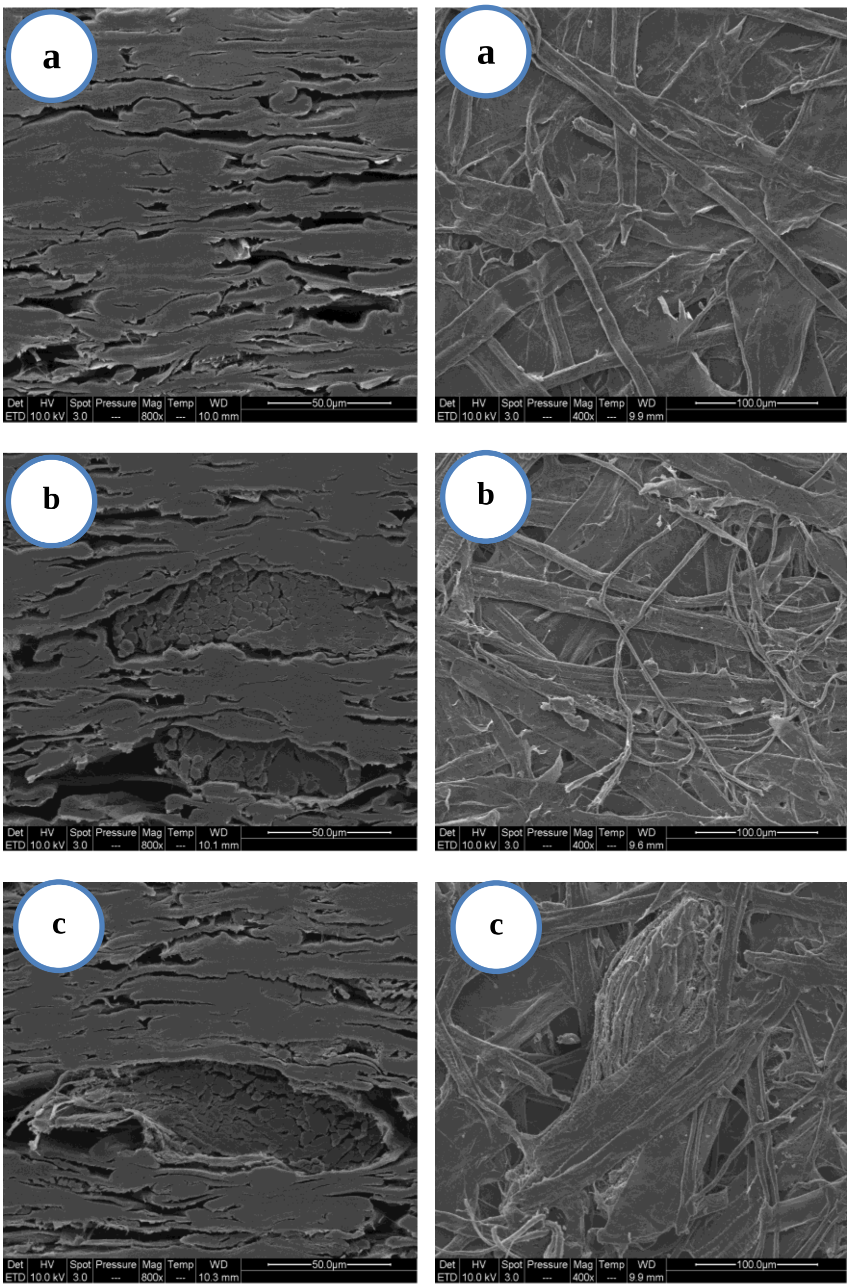

Fig. 1b. SEM images of a) blank carton sheet with fractural and surface image as well as carton sheet with different ratios of treated I, b) 6% and c) 12% with fractural and surface image.

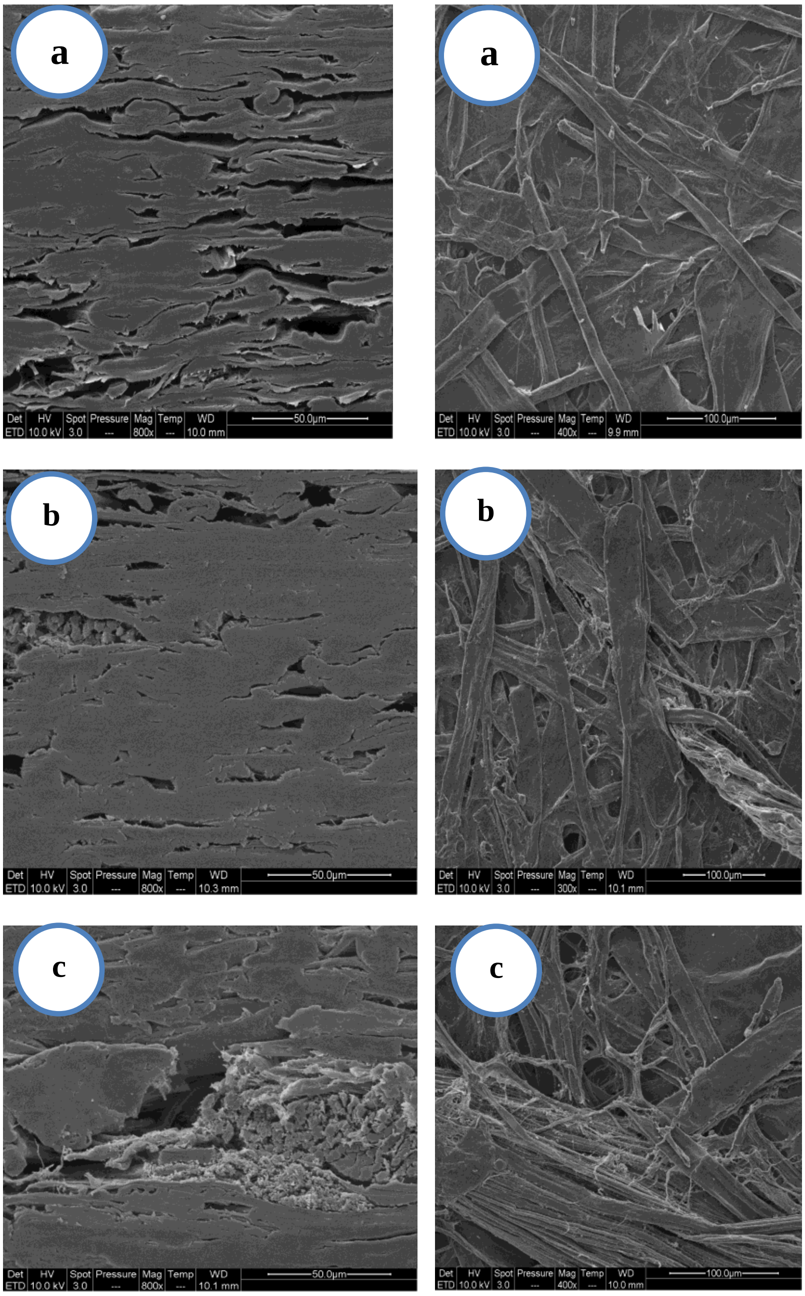

Fig. 1c. SEM images of a) blank carton sheet with fractural and surface image as well as carton sheet with different ratios of treated II, b) 6% and c) 12% with fractural and surface image.

Thermal stability of carton sheets

The Thermal stability of the prepared carton sheets were investigated in details by using thermal gravimetric analysis (TGA). The data of the thermal stability for all samples are presented in Fig. 1 (a- c). Figure 1a shows the result of TGA measurements of blank carton sheet as reference and the carton sheets with different concentrations of untreated leather (6, 9 and 12%). The main weight loss happened from 250 to 370^{o}C. From TGA results, we determine that carton sheets loading with different ratios of untreated leather reveals more thermal stable than the blank carton sheet. The thermal stability increases with the addition of leather by approximately 20 % at 370^{o}C. The enhancement in thermal stability is due to the presence of leather waste nanomaterials and flame retardant materials.

Correspondingly, Fig. 1b revealed the TGA profile of thermal stability of blank carton sheet and the paper sheets treated with different concentrations of treated leather (treated I (6 , 9 and12%)). The major step for weight loss occurred at 240 to 360^{O}C, the untreated carton sheet (blank) displays less thermal stability than treated carton sheet containing diverse concentrations of (treated I). The carton sheet containing 12% treated leather (treated I) represents more thermal stability than the blank carton sheet by 12% at 366°C, while the 6% of treated leather enhanced the thermal stability by nearly 10%. The treated leather using (treated II) which used as filling materials in preparation of carton sheet played a significant role in the thermal stability. It signified more efficiently upon raising the loadings of treated leather (treated II) 12%. This is a typical behavior of the using treated leather (treated I) and the thermal stability increased significantly by increasing the concentration of (treated II) from 6 to 12 % as shown in Fig. 1c.

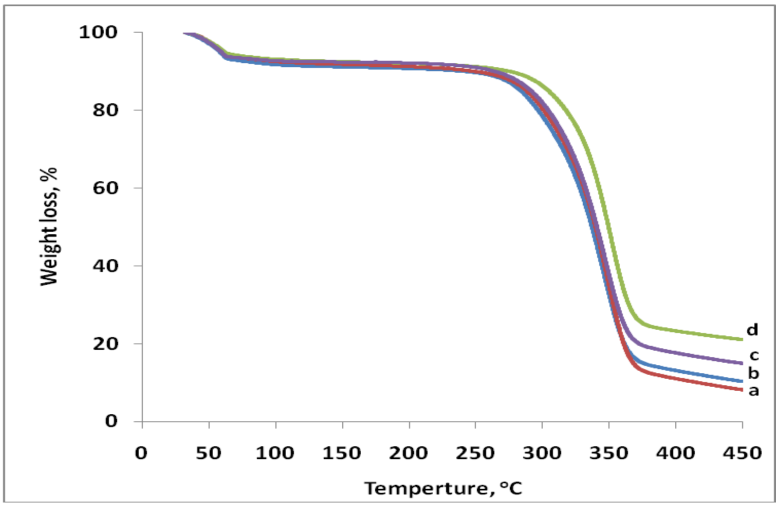

Fig. 2a. TGA of a) blank carton sheet as well as carton sheet with different ratios of unmodified leather, b) 6%, c) 9% and d) 12 %.

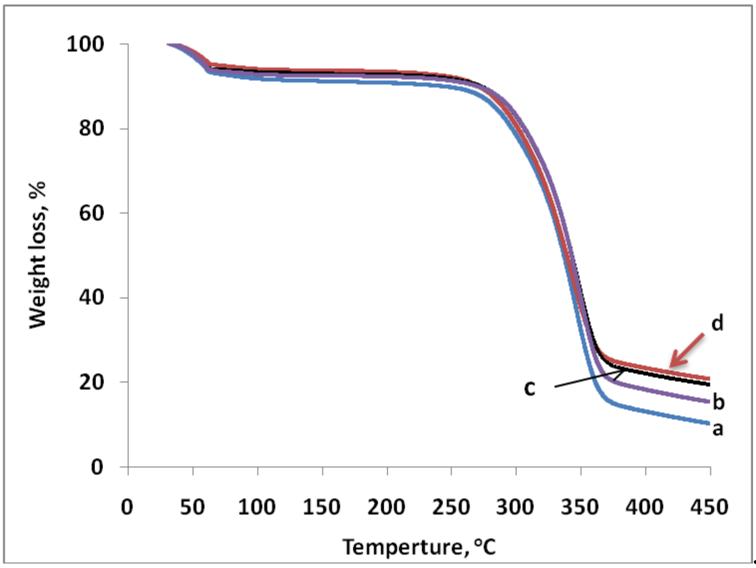

Fig. 2b. TGA of a) blank carton sheet as well as carton sheet with different ratios of treated I b)6 %, c) 9% and d) 12 %.

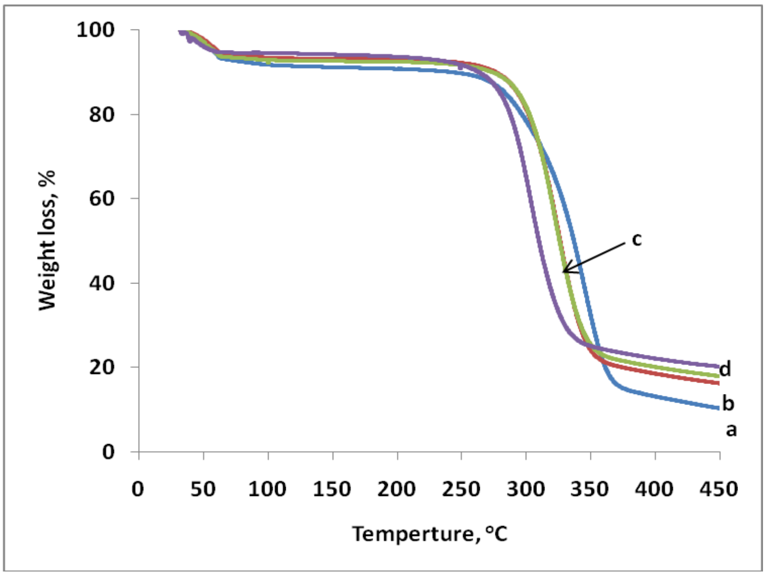

Fig. 2c. TGA of a) blank carton sheet as well as carton sheet with different ratios of treated II, b) 6%, c) 9% and d) 12 %.

Fire Test for the prepared carton paper sheets

From the data presented in Table 4, it is noted that; flame time increase by adding leather waste from 71.25 second for the blank to 90.75 for 12% untreated leather, 83.75 for treated I samples and 78.75 for treated II samples. Also the flame time of burning increase by increasing the ratio of leather waste nanoparticles; this indicates that these nanoparticles help in enhance the flame retardant of paper sheet this is because they are resistant to fire by itself.

The rate of burning for blank was 117.89 mm/min which decrease by increasing addition ratio of nanoparticles leather waste which reach to 92.56 mm/min for untreated leather 100.3 mm/min for treated I and 106 for modified II, this help in reducing spreading of fire through carton sheet. From the results the nanoparticles leather waste succeeded in reducing flame time and rate of burning which can be reflected on fire retardancy of carton sheets.

TABLE 4. The test fire for the prepared carton sheets.

Samples	Flame time (sec)	Burning length (mm)	Rate of burning (mm/min)
Blank	71.25	140	117.89
Unmodified 6%	78.5	140	107.00
Unmodified 9%	90	140	93.33
Unmodified 12%	90.75	140	92.56
Modified I 6%	76.75	140	109.30
Modified I 9%	79.25	140	105.99
Modified I 12%	83.75	140	105.45
Modified II 6%	77.25	140	108.67
Modified I 9%	78.50	140	107.01
Modified I 12%	78.75	140	106.74

Conclusions

This work succeeded to prepare carton sheets loaded with different ratios of waste leather in nanosize, untraeted and treated with flame retardant materials. The results show that the carton sheet loaded with leather waste have high flame retardancy in comparison with blank carton with keeping the other physical and mechanical properties without deterioration, also increasing some properties such as (tensile strentgh, tear factor and air permeability). The results show that nanoparticles leather waste succeeded in reducing flame time and rate of burning which can be reflected on fire retardancy of carton sheets. Delaying firing process will help in protecting the materials from burning, saving many of economic effects. So the prepared paper sheet can be used for different industrial applications especially in packaging applications.

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تحضير كرتون غير قابل للإشتعال بإضافة مخلفات الجلود المحورة

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حجم البقايا الصناعية للجلود كبيرجدا وذو خطورة علي البيئة وأغلبها مواد بروتينية تحتوي علي نسبة من 10-15 % من عنصر الكروم وعند حرق هذه البقايا تحدث تأثيرا سيئا في البيئة المحيطة ولذا فقد عني هذا البحث بمعالجة هذه البقايا بمواد غير قابلة للإشتعال وإضافتها أثناء تحضير ألواح الكرتون وقد تم تقييم هذه الألواح بواسطة الميكروسكوب الإلكتروني بنوعيه وتحديد خواصه الطبيعية والضوئية كما تم اختبار هذه الألواح الإلكتونية كمضادات للإشتعال وثبتت كفاءتها وبذا يمكن التوصية باستخدامها بنجاح كمواد تغليف.