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Recycling Leather Wastes in Paper Industry Aiming to Improvement its Flame Retardant

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ABSTRACT

Wastes extracted from tanning leather industry, were grinded into nanoparticles size and treated with flame retardant materials. The treated leather wastes were then added with different concentrations during paper sheet hand-making. The influence of these leather wastes on flame retardancy and thermal stability of the produced paper sheets were investigated by flame test and Thermal Gravimetric Analysis (TGA). Furthermore, physical properties; permeability, opacity, and brightness and mechanical; tear strength, burst, tensile strength, were evaluated. The surface morphology of the paper sheets was studied using a Scanning Electron Microscope (SEM). We succeeded in producing paper sheets with improved flame retardant based on addition of treated and untreated leather wastes as additives during sheet formation. Furthermore the physical and mechanical properties were unchanged.

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INTRODUCTION

This study tackles two problems: (i) the valorization of industrial waste, here leather industry, (ii) the prevention of fire, or at least the flame retardancy, of paper materials. One of the most significant problems of the leather industry is waste generation: About 60% of leather substance processed in tanneries is rejected, mainly after shaving process, in the form of protein wastes containing about 10–15% chromium (III). These wastes are mainly deposited and burned causing hazards to the environment. In the last few years, chrome shavings as filler in polymer, rubber and paper were studied [1-5]. Improvement of flame retardancy may impact both society and industry preventing fire losses ,in previous study flame retardant of leather were reduced [6] . Worldwide, paper, plastic, and polymers make up a large amount of materials used in everyday life and in many cases they contribute significantly to fire when ignition sources due to their ability to firing [7-8] . In this study, leather wastes were grinded to nanosize, treated with flame retardants, and then added as filler during the paper sheets formation. Using of these wastes help in reduce their hazards and give an economical benefit to paper making and an effective solution for paper firing.

Paper contributes to fire because they have low fire retardancy due to their cellulosic nature while plastic materials are inherently flammable owing to their chemical based upon petrochemical feedstock's [9-12]. Among the pyrolysed components, the most significant is cellulose, which is the principal component in forest species, comprising 41–53% (w/w) of the total weight. The thermal degradation of cellulose takes usually place between 250 and 400 ◦C, through two competing pathways [13-18] : one is the dehydration which leads to char and gases (mainly, CO, CO_2, and H_2O) and the other is the depolymerisation which leads to tar and volatiles through the formation of levoglucosan. Fire retardant is that any substance that by chemical or physical action reduces or inhibits combustion, decreasing thereby both the rate of spread and the fire line intensity of a forest fire. Many studies of retardant effectiveness were carried out based on water solutions containing different chemicals [19-21]. The long-term retardants consist of flame inhibiting chemicals dissolved in water. They remain effective even after water has been removed by evaporation [22].

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The aim of this study is to investigation the effects of the addition of different concentrations nano-sized leather wastes treated with different flame retardant materials on the flammability of paper made from bagasse. Also the physical and mechanical properties of paper sheet were studied.

1. Experimental:

1.1. Materials:

The leather wastes were supplied from medium tannery medium tannery in Misr Alkadima region. The bagasse pulp was supplied by Edfo Company. The triethyl phosphate (TEP) was purchased from "Incorporated-Inc." GmbH (Germany). Bromine was obtained from Sigma-Aldrich.

1.2. Methods

1.2.1. Preparation of the treated leather powder:

Leather waste shavings were disintegrated in a multistage way to obtain a powder, and then sieved through a sieve (0.3 meshes). The resultant leather powder was divided into three parts: (i) untreated leather powder, (ii) leather powder treated with triethyl phosphate (treated I), and (iii) leather powder treated with Bromine (treated II). The treated samples were filtered and dried at 60˚C for 2 hours in an air oven and sieved again through a sieve (0.3 meshes); then they can be included in the preparation of hand sheets.

1.2.2. Preparation of pulp:

*P*ulp samples were impregnated in water for 24 hours, then beated in a Valley beater. The beating process was carried out at 10% pulp consistency at a speed of 150 r.p.m. After beating, the pulp was transferred to a 2 liter measuring cylinder. The stock diluted with water to 2000 mL, disintegrated for 2 minutes at 3000 r.p.m. The pulp was then beaten to reach a value of SR-35 using a Valley beater. Paper sheets were finally prepared.

1.2.3. Hand sheet preparation:

Different amounts (3, 6, 9, and 12 %) of leather wastes, treated and untreated, were added to bagasse pulp suspension at 4 % consistency and stirred for 30 minutes. The mixtures were then diluted to 2% consistency and stirred at 1000 rpm for 30 minutes to ensure homogenous distribution of leather wastes. Paper sheets with mass about (2 gm) 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 60 gm were prepared on a Rapid Khöten sheet former, following the ISO 5269-2 standard method. The hand sheets were conditioned at 23°C and 50 % of relative humidity before testing, as recommended by the ISO 187 standard. Other blank paper sheets were prepared using a hand sheet former from neat bagasse fiber beaten to SR-45 and SR-35, respectively using valley beater.

2. Characterizations:

3.1. 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 (QuantuMike Mitutoyo). Four thickness measurements at different positions were taken on each specimen. The air permeability 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 (Model iso technidyne corporation, New Albany, Indiana, USA).

3.2. 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.

The burst factor was measured according to ISO 1974 standards on a Lhomargy equipment. The tear factor was evaluated on a Lhomargy ED20.

3.3. Flame Test:

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 equal to 45^o. The flame was applied for 2 s at 19 mm from the bottom edge.

3.4. Scanning electron microscope (SEM):

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

3.5. 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^{-1}. The temperature ranged from room temperature up to 500 °C under nitrogen atmosphere.

RESULT AND DISCUSSION

4.1. Basis Weight, thickness and density:

The results of basis weight, thickness and density are presented in Table 1. As we note; density of paper sheet for blank is more than the paper loaded with leather wastes. Density decreased by increasing concentration of untreated and treated leather wastes in the paper sheet.

4.2. Mechanical properties of the prepared paper sheets:

The mechanical properties of the prepared paper sheets, issued from bagasse as cellulosic raw materials and that loaded with different concentrations of untreated leather wastes, as well as treated leather wastes (TEP and Bromine treatments) are shown in Tables 2 and 3. It can be noted that, burst factor increases steadily by increasing the concentration of treated and untreated leather wastes up to 12 % but; it was 133 for blank one while it reach to 170 in case of 12% addition of untreated leather and 135,134 for treated (I) and treated (II), respectively . Also, the tear factor increases by the concentration addition of leather treated or untreated as shown in Table 1; tear factor for blank was 10 and increase and reach to 21 for untreated leather waste and 16 for treated one. This enhancement in burst and tear for paper sheet may be due to intercalation between the cellulosic fibers and leather wastes fiber which reflect on its strength.

Table 3 shows the mechanical properties of paper sheets; the tensile, young modules and elongation%, the tensile of the paper sheet prepared using untreated leather start to increase by the addition of 3% and 6%, they reach a maximum and then decrease by increasing the amount of untreated leather (9% and 12 %). The addition of treated leather during the preparation of paper sheet does not modify the tensile of the prepared paper sheet. On the other hand, the young modules and elongation decreased by the addition of untreated leather or treated leather as shown in Table 3.

4.3. Physical properties of paper sheets:

The brightness and opacity of the prepared paper sheet using untreated leather are displayed in Table 4. Brightness remained unchanged only there is slight increase in case of Leather treated with bromine with 9% and 12% concentration due to white color of bromine. While the opacity of paper sheets increased with increase concentration of treated and untreated leather.

The results for air permeability are presented in Table 5. The air permeability increased gradually by increasing percentage of the addition for both untreated and treated leathers. The air permeability was 135 for blank one and reach to 272 for paper sheet with untreated leather and 245and 169 for treated (I) and treated (II). This increase in air permeability is due to presence of interspecies between leather fiber and cellulosic fiber.

Table 1: Basis weight and thickness of blank paper sheet as well as treated paper sheet

Samples	Weight /Gram Basis Weight ($g.m^{-2}$)	Thickness (µm)	Density
Blank	65.54(±1.68)	95 (±2)	7.2
Untreated			
3%	66.72 (±2.32)	107 (± 1)	6.6
6%	67.23 (±0.85)	115 (± 1)	6.3
9%	87.72 (± 3.13)	124 (±2)	6.3
12%	70.32 (±2.09)	122 (± 1)	5.5
Treated I			
3%	65.77 (±2.93)	102 (± 1)	6
6%	66.18 (±2.12)	109 (±2)	5.8
9%	70.95 (±1.32)	122 (±1)	5.6
12%	71.57(±1.21)	127 (± 1)	5.5
Treated II			
3%	66.33 (±1.13)	104(±2)	5.7
6%	66.78 (±1.27)	120 (± 1)	5.5
9%	69.94 (±1.34)	122 (±2)	5.4
12%	71.57 (±1.32)	125 (±1)	4.8

Table 2: Burst and tear Factor of blank paper sheet as well as treated paper sheet

Paper sheet	Burst index ($kPa \cdot m^2/g$)					Tear index(mNm^2/g)				
	Blank	3%	6%	9%	12%	Blank	3%	6%	9%	12%
Untreated	133 (± 1)	128 (± 1)	130 (± 2)	140 (± 2)	170 (± 1)	10 (±2)	11 (±2)	13 (± 1)	15 (±2)	21 (±2)
Treated I	133 (± 1)	125 (±1)	128 (±1)	130 (±1)	135 (±3)	10 (±2)	10.5 (±1)	11 (±1)	13 (±2)	16 (±2)
Treated II	133 (± 1)	121 (± 1)	127 (± 1)	130 (± 2)	134 (± 2)	10 (± 2)	8 (± 1)	10 (±2)	12 (± 1)	16 (± 1)

Table 3: Tensile, Young modules, and Elongation of paper sheet as well as treated paper sheet

Paper sheet	Tensile index (Nm/g)					Young modules (GPa)					Elongation (mm)				
	Blank	3%	6%	9%	12%	Blank	3%	6%	9%	12%	Blank	3%	6%	9%	12%
Untreated	42.0 (± 1.7)	45.7 (±0.2)	53.0 (±1.4)	38.0 (±1.6)	36.7 (±0.2)	4.4 (±1.4)	4.1 (±0.1)	3.8 (±0.2)	3.7 (±0.1)	2.9 (±0.2)	4.4 (±1.4)	4.7 (±0.2)	4.1 (±0.1)	3.9 (±0.1)	3.6 (±0.1)
Treated I	42.0 (± 1.7)	39.0 (±0.7)	39.2 (±0.5)	40.3 (±0.4)	39.0 (±1.2)	4.4 (±1.4)	3.5 (±0.1)	3.4 (±0.3)	2.8 (±0.1)	3 (±0.1)	4.4 (±1.4)	4 (±0.1)	4.1 (±0.1)	3.9 (±0.2)	3.7 (±0.2)
Treated II	42.0 (± 1.7)	42.0 (±1.9)	41.1 (±0.1)	41.9 (±0.2)	41.7 (±0.1)	4.4 (±1.4)	3.9 (±0.2)	3.2 (±0.2)	3.2 (±0.2)	3.6 (±0.1)	4.4 (±1.4)	4.3 (±0.1)	4.2 (±0.1)	4.1 (±0.1)	4 (±0.1)

Table 4: Brightness and opacity of blank paper sheets as well as treated paper sheets

Samples	% of Filler	Brightness	Opacity
Blank	0.0	60.98 (±0.56)	83.65
Untreated	3%	60.09 (±0.47)	87.26
Untreated	6%	60.59 (±0.19)	90.42
Untreated	9%	60.76 (±0.24)	91.37
Untreated	12%	62.46 (±0.25)	93.21
Treated I	3%	60.73 (±0.66)	86.14
Treated I	6%	60.70 (±0.40)	88.18
Treated I	9%	60.88 (±0.50)	91.31
Treated I	12%	60.85 (±0.26)	92.58
Treated II	3%	60.03 (±0.56)	87.23
Treated II	6%	60.94 (±0.51)	87.98
Treated II	9%	61.25 (±0.39)	90.12
Treated II	12%	61.25 (±0.15)	92.20

Table 5: Air permeability of the prepared paper sheets and treated paper sheet

Samples	% of Filler	Air permeability ($mL.min^{-1}$)
Blank	0.0	135 (± 2.)
Untreated	3%	136 (± 3)
Untreated	6%	161 (±1)
Untreated	9%	223 (±2)
Untreated	12%	272 (± 1)
Treated I	3%	149 (± 1)
Treated I	6%	170 (± 2)
Treated I	9%	178 (± 1)
Treated I	12%	245 (± 4)
Treated II	3%	145 (± 2)
Treated II	6%	172 (± 1)
Treated II	9%	169 (± 1)
Treated II	12%	169 (± 1)

Table 6: Flame retardant and burning length of the paper sheets

Sample	Flame time (s)	Burning Length (mm)
Blank	4	150
Untreated		
3%	6	150
6%	7	150
9%	Not ignited	-
12%	Not ignited	-
Treated I		
3%	6.5	150
6%	7	150
9%	Not ignited	-
12%	Not ignited	-
Treated II		
3%	6	150
6%	Not ignited	-
9%	Not ignited	-
12%	Not ignited	-

4.4. Flame Retardant Test:

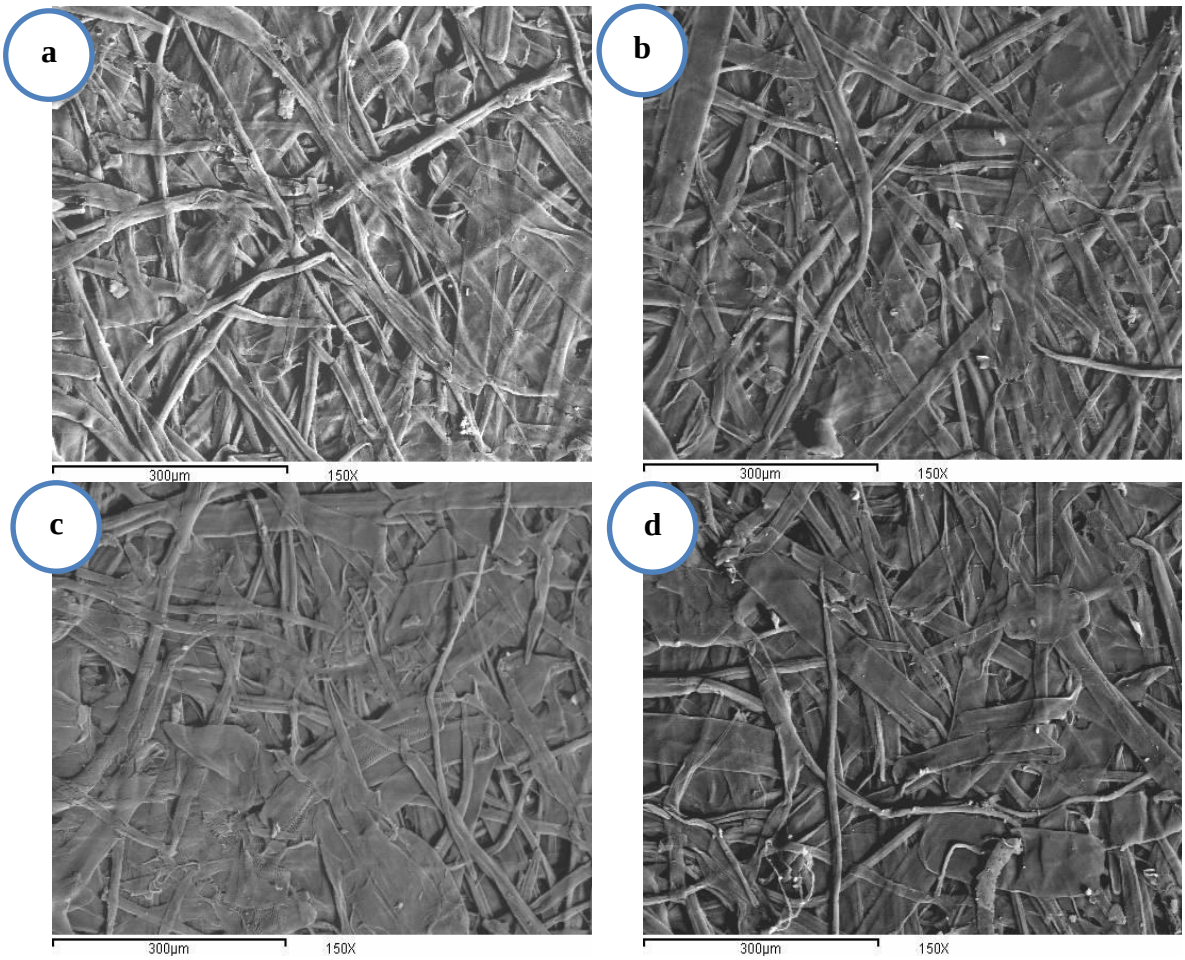
Flame retardant tests for paper sheets; blank and loaded with untreated and treated leather, were carried out. The results are presented in Table 6. Papers loaded with untreated and treated leather with different flame retardants have improved fire retardant than the blank sheet ; the blank one ignites after only 4 seconds, while loaded samples need more time for ignition and by increasing the percentage of loading, papers sheets do not ignited.

Addition of grinded untreated leather waste has obvious effect on flame retardant of paper sheet; by only 9% concentration the paper sheet not ignited. The paper sheet treated with triethyl phosphate (TEP), enhances the resistance to fire gradually. The resistance to fire increases with increase filler content until 9% and 12 % concentration the sample does not ignite. The greatest effect of flame retardant has been seen in the addition of grinded leather wastes with bromine: the sample did not ignite with an addition of only 6%. Physical and mechanical characterizations show that the presence of leather waste treated with TEP and bromine causes only slight decrease of the physical and mechanical properties.

4.5. Scanning electron microscope:

Scanning electron microscope photos for blank paper sheets and paper sheet modified with untreated leather waste are presented in Figure 1a (a-e); (a) for blank and (b, c, e, d) after addition of different concentrations of untreated leather (3, 6, 9, and 12%); respectively . From the Figures, it can be noted that the 'blank' sample has obvious porous are as compared to all other samples. The pore filling increased with increase the concentration of leather particles addition.

Fig. 1b (a-e) display the morphology of the paper sheets containing different ratios of treated leather with TEP : 3, 6, 9, and 12 %. The Figures show the modification of the paper sheet surfaces which become smoother due to leather waste addition which is clogging pores. Also paper sheets modified with leather wastes treated with bromine different concentrations, were shown in Fig. 1c (a-e), the filler particles are situated in the paper sheet pores and appear as more homogenous and smooth surface.



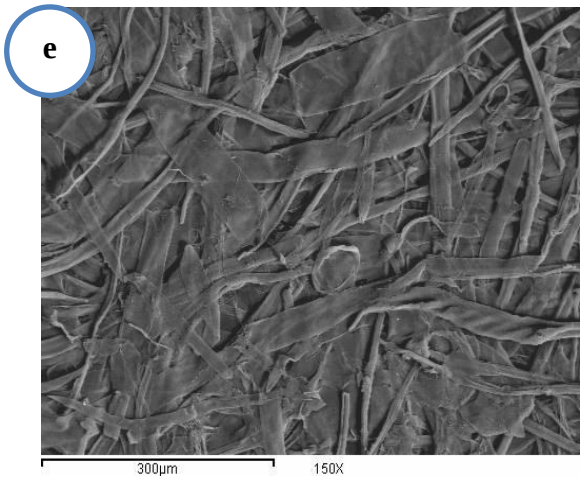
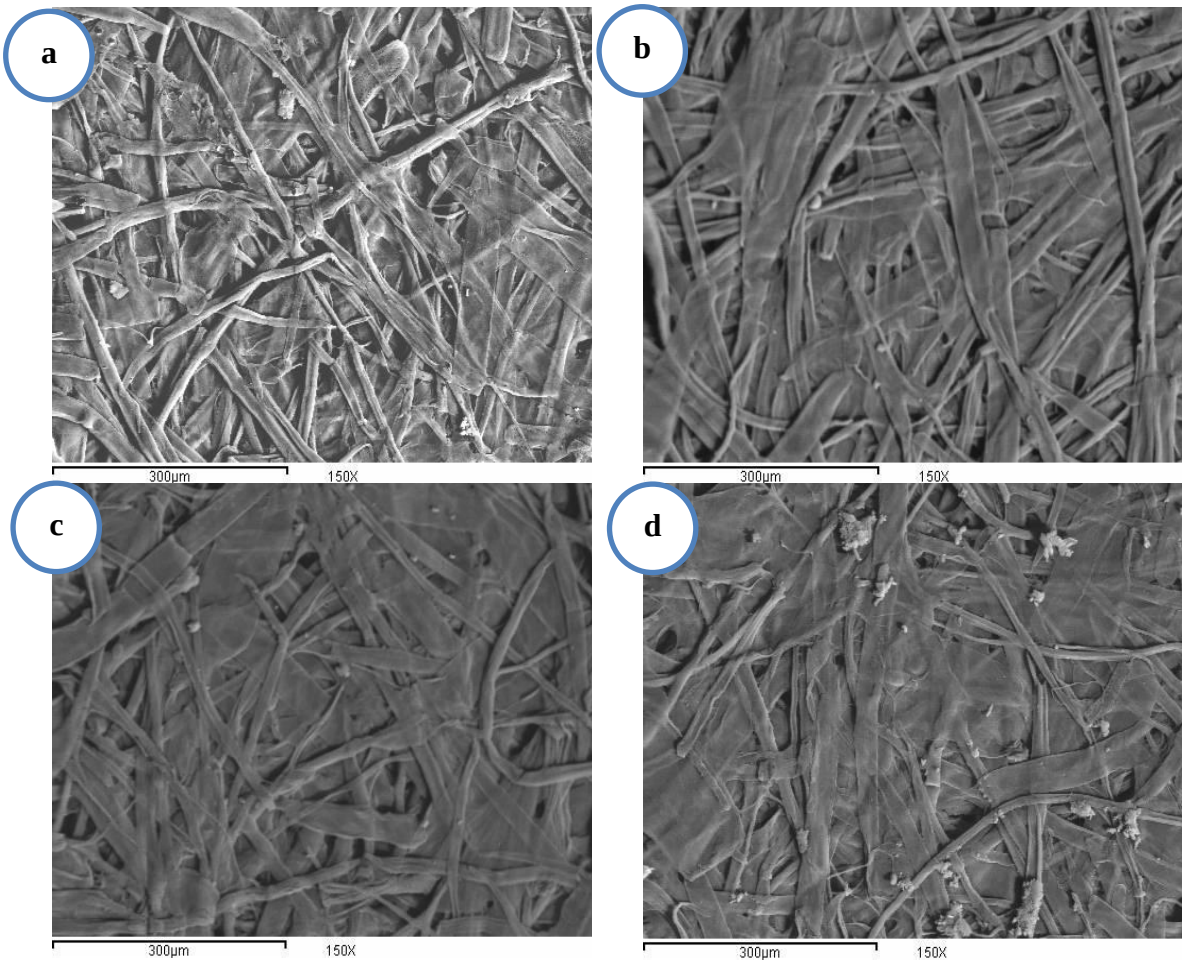


Fig. 1a: SEM images of a) Blank paper sheet as well as paper sheet with different ratios of unmodified leather, b) 3%, c) 6%, d) 9%, and e) 12%



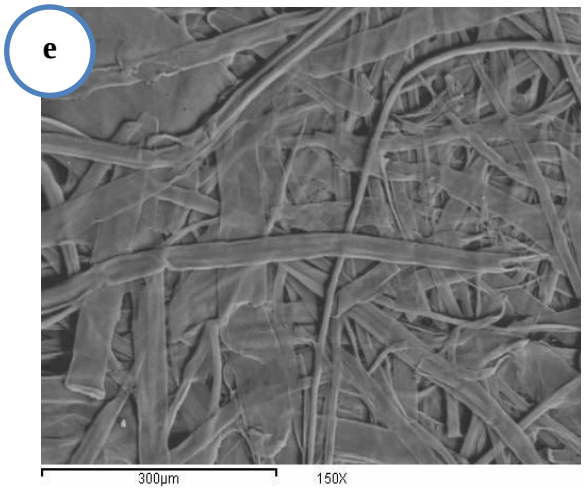
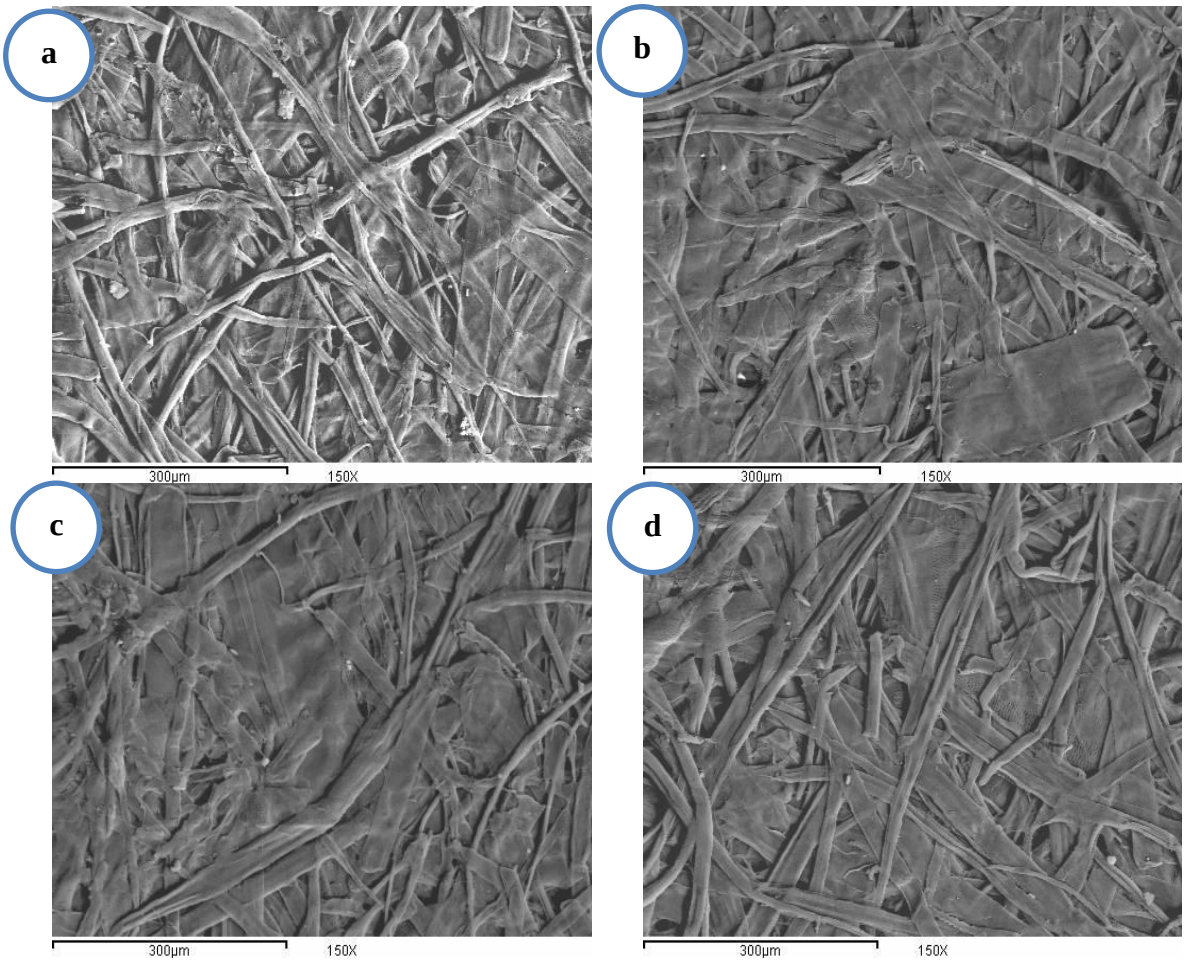


Fig. 1b: SEM images of a) Blank paper sheet as well as paper sheet with different ratios of treated I, b) 3%, c) 6%, d) 9%, e) 12%



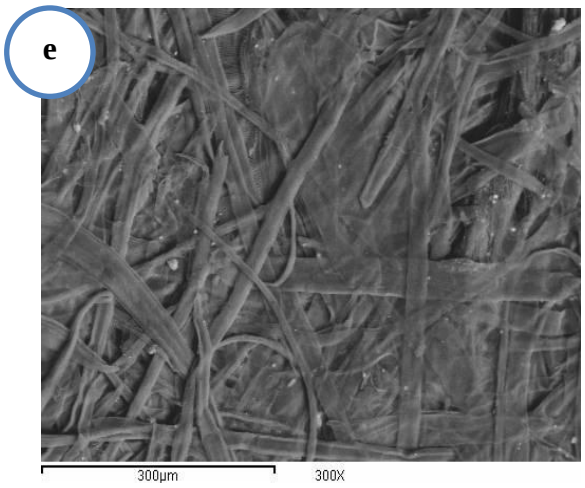


Fig. 1c: SEM images of Blank paper sheet (a) as well as paper sheet with different ratios of treated II, b) 3%, c) 6%, d) 9%, e) 12%

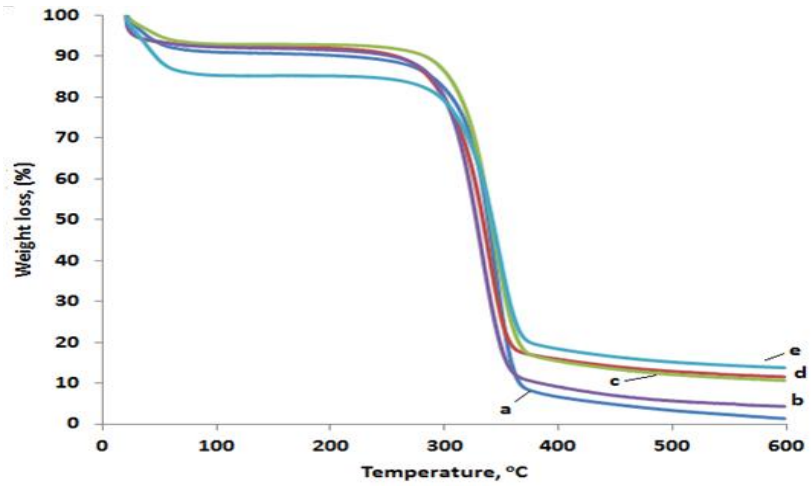


Fig. 2a: TGA of a) paper sheet, b) 3% untreated leather, c) 6% untreated leather, d) 9% untreated leather, e) 12% untreated leather

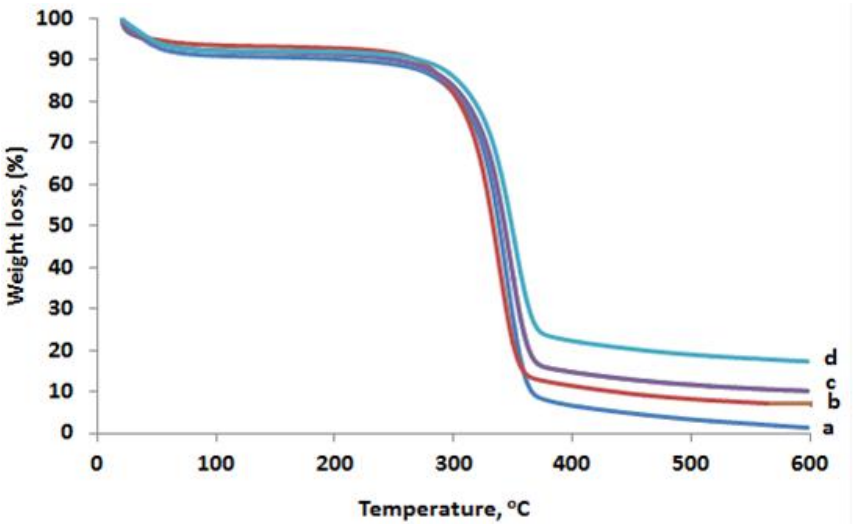


Fig. 2b: TGA of a) paper sheet as well as, b) 3% treated I, c) 6% treated I, d) 12% treated I

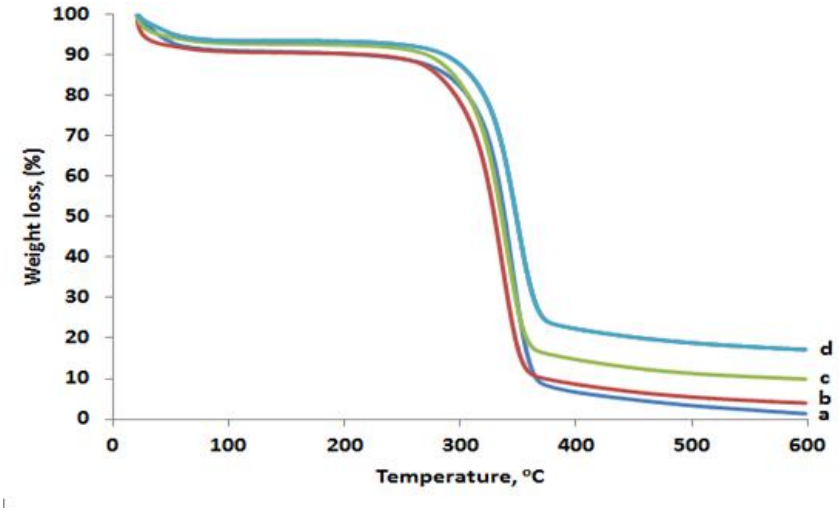


Fig. 2c: TGA of a) paper sheet as well as, b) 3% treated II, c) 6% treated II, d) 12% treated II

4.6. Thermal gravimetric analysis:

The data of the thermal stability for all samples are presented in Figure 2 (a- e). Figure 2a shows the result of TGA measurements of blank paper sheet as the reference, and (b, c, d, e) is the TGA of the paper sheets with different concentrations of untreated leather (3, 6, 9 and 12%). It was found that; the main weight loss appeared between 280 and 380^{o}C. From TGA results, we conclude that paper sheets loaded with untreated leather are more thermal stable than the blank paper sheet. The thermal stability increases with the addition of leather by about 15% at 380°C. The enhancement in thermal stability is due to the presence of leather waste nanomaterials and flame retardant materials.

Figure 2b revealed the TGA profile of thermal stability of blank paper sheet and the paper sheets treated with various concentrations of TEP (treated I), (3, 6 and12%). The main step for massive weight loss occurred between 280 and 380°C. The sheet containing 12% treated leather (I) reveals more thermal stability than the blank paper sheet by 20% at 380°C. The 6% of treated leather enhanced the thermal stability by nearly 10%. The treated leather using bromine (treated II) which was used as filling materials in the preparation of paper sheet played a pronounced role in the thermal stability. It acted more effectively upon increasing the concentration of treated leather (treatment II) up to a value of 12%. This result is a typical behavior of the used treated leather (treatment I). The thermal stability increased significantly by increasing the concentration of (treated II) from 3% to 12 % as shown in Fig. 2c.

The paper sheets loaded with leather wastes treated or untreated have more thermal stability than blank one.

Conclusions:

This study aimed to produce paper sheet having flame retardant properties using relatively inexpensive nanoparticles issued from leather wastes. The leather wastes may also be treated by TEP and bromine and used at different concentrations during sheet making. These addition lead to double the time needed for ignition. This time increase is associated with the thermal analysis and lower flammability. Moreover, papers did not ignite by using 9% and 12% TEP and Bromine. Neither the mechanical properties, nor the optical properties were affected by the addition of the treated leather wastes. The scanning electron microscope showed the clogging of surface pores of the paper sheets which reflect on smoothness of paper sheets. These results showed that the addition of leather wastes has improved significantly the flammability properties. This approach can be also extended to various fields of chemistry such as polymers and rubbers.

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