Sustainability of Paper & Sugar Industries via Molasses: Novel Green Nanocomposites from Upgraded Recycled Cellulose Fibers

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Abstract: The present work leads to sustainability (responsible management of resources consumption) of both paper and sugar industries. It, simultaneously, upgrades *recycled waste paper (namely old newsprint)* and creates a new use for *molasses* (*an important byproduct of the sugar industry*). This study introduces -for the first time world wide- a novel environmentally safe approach to upgrade recycled natural cellulose fibers (*waste paper namely old newsprint*), for use as specialty paper green nanocomposites suitable for several advanced purposes. The recycled cellulose fibers are upgraded by increasing their alpha cellulose content, and restoring their natural nanoporous structure, which is -normally- collapsed due to the first cycle of papermaking. *Molasses* is then incorporated into this restored nanoporous structure to obtain paper green nanocomposites filled with kaolin in presence of *molasses*. In comparison to conventionally recycled waste paper, the green paper nanocomposites -produced in this work- exhibit high dry and wet strength, and a surprisingly tremendous retention of inorganic fillers used in papermaking.

This was achieved through a green nanotechnology process, where the solvent used is water (the ideal green solvent). The input and output components are renewable environmentally safe materials i.e. *waste paper* (cellulose fibers) and *molasses* (a by product of the sugar industry). The procedures used are simple physical processes conducted at room temperature rather than hazardous chemical reactions. Thus, saving energy, and decreasing the risk of chemical and thermal accidents, pollutive releases, explosions, and fires.

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1. Introduction

Recently, the authors[1-8] introduced fully green nanotechnology as a gateway to beneficiation of natural cellulose fibers. Fully green nanotechnology is the *ideal case* of green nanotechnology. If a fully green nanotechnology process should involve a solvent, this solvent should -ideally- be water (the most important green solvent). Moreover, all the input and output components (materials), involved in fully green nanotechnology, should be environmentally safe. (e.g. polysaccharides and natural cellulose fibers). The procedures, involved in fully green nanotechnology, should -preferably- be simple physical or biological processes rather than hazardous chemical reactions. These fully green processes should be performed at low temperatures -ideally- room temperature. Thus, saving energy, and decreasing the risk of chemical and thermal accidents, pollutive releases, explosions, and fires. A perfect example of fully green nanotechnology, is the manipulation of the natural nanoporous structure of cellulose fibers to increase the water absorption and reactivity of cellulose fibers, to produce water absorbent paper nanocomposites, or to greatly increase the strength of paper made from cellulose fibers. This was achieved by incorporating the natural nanoporous structure, of water swollen cellulose fibers, by the nanoadditives sucrose and glucose.[1-8]

It is worth mentioning that Nanoporous materials are classified as naturally occurring nanoporous materials, and synthetic nanoporous materials. Naturally occurring nanoporous materials may be of mineral origin (such as zeolites), or of biological origin (such as the nanoporous structure of the cell walls of cellulose fibers).[4, 9, 10]

Sucrose and glucose molecules are entrapped in the cell wall nanopores of cellulose fibers, during the collapse of these pores, as the fibers are dried. The sucrose and glucose molecules act as spacers, and prevent the irreversible collapse of the natural nanoporous structure of cellulose fibers, which – normally – occurs during drying. Thus the incorporation of sucrose or glucose into cellulose fibers leads to nanocomposites of increased water uptake (water retention value), and increased reactivity (i.e. increased accessibility to reagents). It was also shown that incorporating the nanoporous structure of cellulose fibers, with sucrose, leads to paper nanocomposites of enhanced strength (breaking length). The cell walls, on both sides of the incorporated sucrose spacers, are stressed during drying because sucrose spacers hinder them to relax.

This leads to a strain, which makes some microfibrils partially released and protrude out of the fibers. This in turn leads to more efficient entanglement of the fibers, and hence increases the strength of the prepared paper nanocomposites. In other words, a sort of fibers beating takes place. The authors called this phenomenon *incorporation beating* to differentiate it from chemical and mechanical beatings, conventionally applied to increase the strength of paper.[1]

Recently Fahmy & Mobarak succeeded -for the first time- to manipulate the strength promoting effect of sucrose as a means to counteract the ultimate fate of deterioration in strength of paper, due to addition of inorganic fillers such as kaolin.[4] Moreover, sucrose was proven -for the first time- to act as retention aid for inorganic fillers such as kaolin. The authors called this phenomenon *incorporation retention* to differentiate it from the conventional types of retention of inorganic fillers. To achieve these aims, the authors prepared an advanced paper nanocomposite involving two additives -a nanoadditive and a conventional additive- within a matrix of natural cellulose fibers. The first additive (the nanoadditive) is sucrose, which incorporates the nanoporous structure of the cell walls of cellulose fibers. The second additive (the conventional additive) is kaolin, the famous paper filler. Kaolin is enmeshed between the adjacent cellulose fibers. This advanced paper nanocomposite was prepared by simple techniques.[4]

It is worth mentioning that when aqueous solutions of sucrose are equilibrated with the water-swollen pulp (cellulose fibers), sucrose should be able to penetrate into every micropore or nanopore larger than 8 A˚ (0.8 nm). The volume of these sucrose-accessible pores amounts to 86.5% of the total pore volume of the micropores. Thus, the dissolved sucrose molecules should be distributed rather uniformly throughout the fiber cell wall, except for pores less than 8 A˚ in size. These calculations are based on the solute exclusion data of Stone and Scallan and the size of the sucrose molecules derived from them.[11]

By mixing water-soluble and water insoluble polysaccharides, the insoluble ones could act as matrix while the soluble ones work as reinforcing part. As composites may contain, except polymeric components, also water soluble/insoluble compounds, polysaccharide swelling in water and water sorption is beneficial in relation to applications like drug-delivery, paper toils or similar possibilities. Combination of natural cellulose fiber with kaolin in the presence of sucrose results in fully green nanocomposites.[4, 12]

The successful results, obtained due to using sucrose and glucose as additives in papermaking, encouraged the author to expand the studies -for the first time- to a sugar industry byproduct rich in sucrose, which is molasses.

Fahmy introduced molasses -for the first time worldwide- as a new additive in papermaking.[2, 3] Using this byproduct, as an additive for cellulose fibers, succeeded in producing paper nanocomposites of enhanced dry and wet strength and improved water absorbance.[2, 3]

Furthermore, paper nanocomposites -involving kaolin, molasses, and virgin high alphacellulose natural wood fibers- retained larger amounts of kaolin while exhibiting greater strength, as compared to their molasses-free counterparts. Thus, molasses was shown to be a nano-based master cheap environmentally safe retention aid and strength promoter in papermaking.[6]

Moreover, using gums (including starch) as additives in papermaking enhances the strength of paper.[14] Molasses contains both sucrose and gums (including starch). Molasses is a byproduct of sugar industry, which is cheaper than sucrose; and a major part of sucrose lost in sugar industry resides in molasses. Therefore, molasses was chosen as a new additive in papermaking.[2, 3]

Molasses is obtained during the sugar-extraction process. The liquid discharged by the centrifugals in the last stage of sugarcane juice processing, after no more sugar can be separated from the sugarcane juice by usual factory methods, is called final molasses. Molasses contains sucrose, which cannot be recovered by economic methods. Sucrose (lost in molasses) represents the highest proportion of the losses incurred in the processing of sugarcane. This loss may reach about 9% of the total sucrose. Thus sucrose lost in molasses is a major consideration, and is the principal reason for the varied and extensive inquiries which have been conducted on the profitable utilization of this valuable byproduct. The sucrose content in molasses may range from about 32% - 44%. In addition to sucrose, reducing sugars are present in molasses, namely glucose and fructose. The content of reducing sugars ranges from about 10 to 15 percent. Thus the principal value of molasses as an industrial raw material lies in its content of fermentable sugars, which amounts to about 50% by weight. Gums (including starch) are also present in molasses. The content of gums (including starch) ranges from about 3 to 5 percent by weight.[13]

Considering paper industry, *old newsprint* is one of the famous cellulosic fibers available as waste paper. Old newsprint is an abundant cellulosic raw material, which is cheaper than virgin wood pulps. Due to the concern about environmental issues and economical aspects, the importance of recycling and utilization of secondary fibers has been recognized.

Nowadays, around 50% of the total produced paper is recovered.[15, 16] Generally, it can be stated that pulp properties of recycled cellulose fibers decline during the reprocessing. Deterioration of recycled fiber properties is mainly due to the irreversible changes occurring in fiber structure caused by repeated chemical and mechanical treatments and drying.[17] Traditional methods for increasing the strength of recycled paper involve either beating of the fibers or adding dry strength additives.[18]

Our successful basic studies on green nanotechnology, the results obtained -in cases of biological never dried cotton fibers, high alpha cellulose cotton linters, and viscose wood pulps- called for trials to upgrade recycled old newsprint.

The present study introduces, for the first time world wide, a novel environmentally safe approach to upgrading recycled natural cellulose fibers, for use as specialty paper green nanocomposites suitable for several advanced purposes.

It is planned to make all-in-one by applying simple green nanotechnology through treatment of the recycled old newsprint by NaOH at room temperature for short time under specific conditions. This treatment intends to increase the high alpha cellulose content of recycled cellulose fibers. It, also, intends to restore the natural nanoporous structure of cellulose fibers.[1] This natural nanoporous structure is -normally- collapsed in case of old fibers of waste paper due to the first cycle of papermaking.

In this work, the prepared high alpha cellulose recycled old newsprint is planned to be investigated in the non-dried water-swollen state -after treatment with NaOH. Thus, its restored nanoporous structure will be manipulated -by incorporating it with molasses- to obtain specialty paper green nanocomposites filled with kaolin in presence of molasses (the nano-based master cheap environmentally safe retention aid and strength promoter, which we have recently introduced as a new additive in papermaking).[2, 3, 6]

The present work achieves sustainability (responsible management of resources consumption) of both the paper and sugar industries. It creates a new use for molasses (an important byproduct of the sugar industry) and simultaneously upgrades recycled waste paper.

2. Material and Methods: -

Waste paper used: The waste paper used in this work was composed of uncolored and colored newsprints in equal ratios.

The conventional additive (inorganic filler kaolin) used in this work was Egyptian upgraded kaolin prepared on pilot scale, kindly provided by Metallurgical Research and Development Institute, El-Tebeen, Egypt. Its specifications and analyses are: Kaolinite 92.43% (Al_2O_3 35.21%, total SiO_2 44.43%, Fe_2O_3 0.92%, TiO_2 1.38%, moisture content 0.73%, ash content 87.99%, and brightness 73.90%). The bulk density of this kaolin was 0.846 before grinding and 1.1813 after grinding.

2.1. Deinking and recycling of old newsprint:

An efficient deinking and recycling method for old colored newsprint was implemented. This method was previously established by the authors and others.[19-21] Analyses of the deinked recycled newsprint are shown in Table 1.

2.2. Treatment of non-dried deinked recycled newsprint by NaOH:

The non-dried deinked recycled newsprint was treated by 18% NaOH for half an hour at room temperature, then washed till neutrality and kept non-dried ready for papermaking. We have carried out chemical and physical analyses of the produced high alpha cellulose recycled old newsprint. The results of the analyses and physical properties are reported in **Table 2**.

2.3. Filling the cellulose fibers (recycled upgraded old newsprint fibers) with the conventional additive (inorganic filler kaolin): -

In all experiments, the cellulosic fibers were mixed with kaolin and beaten for 15 minutes. The consistency was adjusted to 6%. The fibers were filled with increasing kaolin quantities (5, 10, 15 and 20g of kaolin per 100g of pulp fibers).

2.4. Incorporating the nanoadditive (molasses) into the nanoporous structure of cell walls of kaolin-filled cellulose fibers (recycled upgraded old newsprint fibers): -

The incorporation methods used in the present work were recently established by the authors.[1-7] After several preliminary experiments, we fixed the optimum conditions for manipulating the nanoadditive (molasses) as a retention aid and strength promoter. The beaten non-dried kaolin-filled fibers were incorporated with molasses solution of the concentration 10 % w/w, and stirred in the mixer for 15 minutes.

2.5. Paper Sheet Making: -

Paper sheet composites were made from fibers filled with kaolin only, and paper sheet nanocomposites were made from molasses-incorporated kaolin-filled fibers. The paper sheets were prepared according to the SCA standard, using the SCA - model sheet former (AB Lorenzen and Wetter).

2.6. Determination of the retention value of the inorganic filler kaolin: -

The amounts of the inorganic filler kaolin, retained in the kaolin-filled paper sheet composites and in the molasses-incorporated kaolin-filled paper sheet nanocomposites, were determined by ignition of accurately weighed paper sheets. The retention value was calculated as the ratio of the amount of filler retained in the paper sheet to that originally added. The loss resulting from filler dehydration due to ignition was taken into consideration.[22-24] The retention value was calculated by the formula:

Retention Value % = (wt of retained kaolin/wt of added amount of kaolin) X 100

3. Results and Discussion: -

3.1. Effect of Filling Upgraded Recycled Cellulose Fibers (high alpha cellulose recycled newsprint) with the Conventional Additive (Inorganic Filler Kaolin), in Absence of Molasses: -

Table 3 shows the properties of paper composites made from recycled cellulose fibers i.e. high alpha cellulose recycled newsprint, filled with increasing amounts of kaolin (5, 10, 15 and 20g of kaolin per 100g of fibers).

It is evident from **Table 3** that the strength (breaking length) of the paper composites decreased with increasing the amount of added kaolin. The breaking length of the blank (kaolin-free paper) was 3015 m, while that of the kaolin-filled paper composites decreased to 2306 m, due to addition of 20g of kaolin per 100g of recycled fibers. Thus the percentage decrease in breaking length, due to addition of kaolin, reached 20.20 %.

Also, the wet breaking length of paper composites decreased due to addition of kaolin. The wet breaking length of the blank (kaolin-free paper) was 880 m, while that of the kaolin-filled paper composites decreased to 605 m, due to addition of 20g of kaolin per 100g of recycled fibers. Thus the percentage decrease in wet breaking length, due to addition of kaolin, reached about 31.25%.

This decrease in strength (breaking length) of the paper composites is a normal phenomenon, observed due to addition of inorganic fillers such as kaolin. These fillers are enmeshed between the adjacent cellulose fibers, and hence interrupt the inter-fiber bonding between adjacent fibers.[14, 22-25]

3.2. Effect of Incorporating the Kaolin-Filled Upgraded Recycled Cellulose Fibers (high alpha cellulose recycled newsprint) with the Nanoadditive (Molasses) on the Properties of the Produced Advanced Paper Nanocomposites : -

In these experiments, the beaten non-dried kaolin-filled upgraded recycled cellulose fibers (high alpha cellulose recycled newsprint) were incorporated with molasses solution of the concentration 10 %w/w. Paper sheet green nanocomposites were prepared from these beaten non-dried kaolin-filled molasses-incorporated fibers, as mentioned in the experimental part.

Table 4 shows the properties of paper green nanocomposites made from the molasses incorporated kaolin-filled upgraded recycled cellulose fibers (high alpha cellulose recycled newsprint), at increasing amounts of kaolin, of 5, 10, 15 and 20g per 100g of fibers.

It is evident by **comparing Table 4** and **Table 3** that the breaking length of paper green nanocomposites, produced from molasses-incorporated kaolin-filled fibers, is greater than that of paper composites produced from the kaolin-filled molasses-free fibers. This is true for all the added amounts of kaolin. At addition of 20g of kaolin per 100g of recycled fibers, the breaking length of the kaolin-filled molasses-free paper composites was 2406 m, while that of the molasses-incorporated kaolin-filled paper nanocomposites was 3600 m. Thus, there is a percentage increase of 49.63% in the breaking length, due to incorporation of the cellulose fibers by molasses.

It is evident from **Table 4** that the dry breaking length of the molasses-incorporated kaolin-filled paper green nanocomposites, even, surpassed the dry breaking length of the blank (kaolin-free paper). This was true for all the added amounts of kaolin. Even at the highest amount of added kaolin (20g per 100g of fibers), the dry breaking length of the molasses-incorporated kaolin-filled paper nanocomposites was greater, by about 19.40%, than that of the blank kaolin-free paper.

The wet breaking length of paper green nanocomposites, produced from molasses incorporated kaolin-filled fibers, was greater than that of paper composites produced from the kaolin-filled molasses-free fibers **(compare Table 3 and Table 4)**. This is true for all the added amounts of kaolin. At addition of 20g of kaolin per 100g of fibers, the wet breaking length of the kaolin-filled molasses-free paper composites was 605 m, while that of the molasses-incorporated kaolin-filled paper green nanocomposites was 1068 m. Thus, incorporation of the cellulose fibers, with molasses, led to a percentage increase of 52.61% in the wet breaking length.

Table 4 shows that the wet breaking length of the molasses-incorporated kaolin-filled paper green nanocomposites, even, surpassed the wet breaking length of the blank (kaolin-free paper). This was true for all the added amounts of kaolin. Even at the highest amount of added kaolin (20g per 100g of fibers), the wet breaking length of the molasses-incorporated kaolin-filled paper green nanocomposites was greater, by about 21.36%, than that of the blank kaolin-free paper.

It is clear from these results that incorporating cellulose fibers, with molasses, succeeded in counteracting the deterioration in strength of paper, that occurs due to addition of inorganic fillers such as kaolin. Sucrose -present in molasses- acted as a strength promoter in the paper nanocomposites, produced from the molasses-incorporated kaolin-filled fibers. The strength (breaking length) of these paper nanocomposites, even, surpassed that of the blank (filler-free paper). Incorporating cellulose fibers, with sucrose, leads to incorporation beating of the fibers, and thus increases the strength of the produced paper nanocomposites.

Moreover, the gums and starch -present in molasses- exerted an additional strength promoting effect.

3.3. Manipulating the Nanoadditive (Molasses) for Upgrading the ability of Recycled Cellulose Fibers to retain Inorganic Fillers Such as Kaolin:-

Table 5 shows the retention value of kaolin for both the kaolin-filled molasses-free paper composites, and the kaolin-filled molasses incorporated paper nanocomposites.

It is evident from **Table 5** that incorporation of cellulose fibers, by molasses, resulted in an increase in the amount of kaolin retained in the produced paper nanocomposites, relative to the case of the molasses-free kaolin-filled paper composites. This was true for all the added amounts of kaolin. There was a percentage increase of about 195.16% in the retention value of kaolin, due to incorporation of the cellulose fibers by molasses (at added amount of kaolin of 15 g per 100g of fibers). The retention value of kaolin in the case of molasses-free kaolin-filled paper composites was 28.90%, however, it increased tremendously to 85.30% in the case of the molasses-incorporated kaolin-filled paper nanocomposites.

Table 1: Analysis and physical properties of the deinked recycled old newsprint

Alphacellulose %		65.80
Hemicellulose %		15.00
Lignin%		16.21
Ash%		1.73
Water retention value (WRV) %	Non-dried	88.40
	Air-dried	78.00

Table 2: Analysis and physical properties of the produced upgraded recycled high alpha cellulose old newsprint

Alphacellulose %		90.71
Hemicellulose %		7.51
Ash%		1.00
Water retention value (WRV) %	Non-dried	109.75
	Air-dried	83.42

Table 3: Effect of filling the upgraded recycled cellulose fibers (high alpha cellulose recycled newsprint) with the conventional additive (inorganic filler kaolin) -in absence of molasses- on the properties of the produced paper composites

Amounts of the added kaolin (in grams per 100 grams of recycled fibers)	zero	5	10	15	20
Breaking length in meters	3015	2822	2700	2515	2406
% decrease in breaking length	----	6.40	10.45	16.58	20.20
Wet breaking length in meters	880	705	621	611	605
% decrease in wet breaking length	----	19.88	29.43	30.57	31.25

Table 4: Effect of incorporating the kaolin-filled Upgraded Recycled Cellulose Fibers (high alpha cellulose recycled newsprint) with nanoadditive (molasses) on the properties of the produced advanced paper green nanocomposites

Amounts of the added kaolin (in grams per 100 grams of fibers)	zero	5	10	15	20
Breaking length in meters	3015	3620	3612	3605	3600
% increase in breaking length	----	20.07	19.80	19.57	19.40
Wet breaking length in meters	880	1075	1061	1065	1068
% increase in wet breaking length	----	22.16	20.57	21.02	21.36

Table 5: Manipulating the nanoadditive (molasses) for upgrading the ability of recycled cellulose fibers to retain the inorganic filler kaolin

Amounts of the added kaolin (in grams per 100 grams of recycled fibers)	zero	5	10	15	20
Retention value of kaolin in case of paper composites produced from kaolin-filled molasses-free recycled fibers %	----	30.20	29.66	28.90	29.14
Retention value of kaolin in case of paper nanocomposites produced from molasses-incorporated kaolin-filled recycled fibers %	----	81.22	83.50	85.30	86.71
Percentage increase in the retention value of kaolin, due to molasses-incorporation into the kaolin-filled recycled fibers %	----	168.94	181.52	195.16	197.56

These results show clearly that sucrose -present in molasses- acts as a retention aid for inorganic fillers such as kaolin. It is assumed that, during paper sheet formation, sucrose decreases the collapse of the nanoporous structure of the fibers. This collapse -normally- takes place at paper sheet formation, due to drying of the fibers. Thus, during paper sheet formation, the sucrose-incorporated fibers are more swollen and thicker, relative to the sucrose-free fibers. This fiber swelling decreases the size of the gaps present between the fibers, during paper sheet formation. Therefore, lesser amount of inorganic filler can escape through these narrowed gaps, during the water drainage, which occurs at paper sheet formation. Eventually, more inorganic filler is enmeshed between these swollen thickened sucrose-incorporated fibers. We called this type of retention ***"incorporation retention"*** to differentiate it from the conventional types of retention of inorganic fillers.

Moreover, the gums and starch -present in molasses- exerted an additional retention aiding effect.

Conclusions

The present work enhances the sustainability (responsible management of resources consumption) of both the paper and sugar industries. It creates a new use for ***molasses*** (***an important byproduct of the sugar industry***) and simultaneously ***upgrades recycled waste paper (namely old newsprint)***.

This study introduced -for the first time worldwide- a novel environmentally safe approach to upgrade recycled natural cellulose fibers (*waste paper namely old newsprint*). The recycled cellulose fibers were upgraded by increasing their alpha cellulose content, and restoring their natural nanoporous structure, which is -normally- collapsed due to the first cycle of papermaking.

A simple green nanotechnology process was adopted through treatment of the recycled cellulose fibers by NaOH at room temperature for short time under specific conditions. This treatment increases the alpha cellulose content of recycled cellulose fibers from 65.80% to 90.71%, and restores the natural nanoporous structure of cellulose fibers. To make use of the restored nanoporous structure, the upgraded high alphacellulose recycled old newsprint was further processed -after treatment with NaOH- while it was still in the *non-dried water-swollen state*.

Molasses was incorporated into this *non-dried water-swollen* restored nanoporous structure to obtain paper green nanocomposites filled with kaolin in presence of ***molasses***.

In comparison to conventionally recycled waste paper, the green paper nanocomposites -produced in this work- exhibit high dry and wet strength, and a surprisingly tremendous retention of inorganic fillers used in papermaking.

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