Upgrading Recycled Paper Using *Astragalus gossypinus* Tragacanth Gum as a Bio-based Additive

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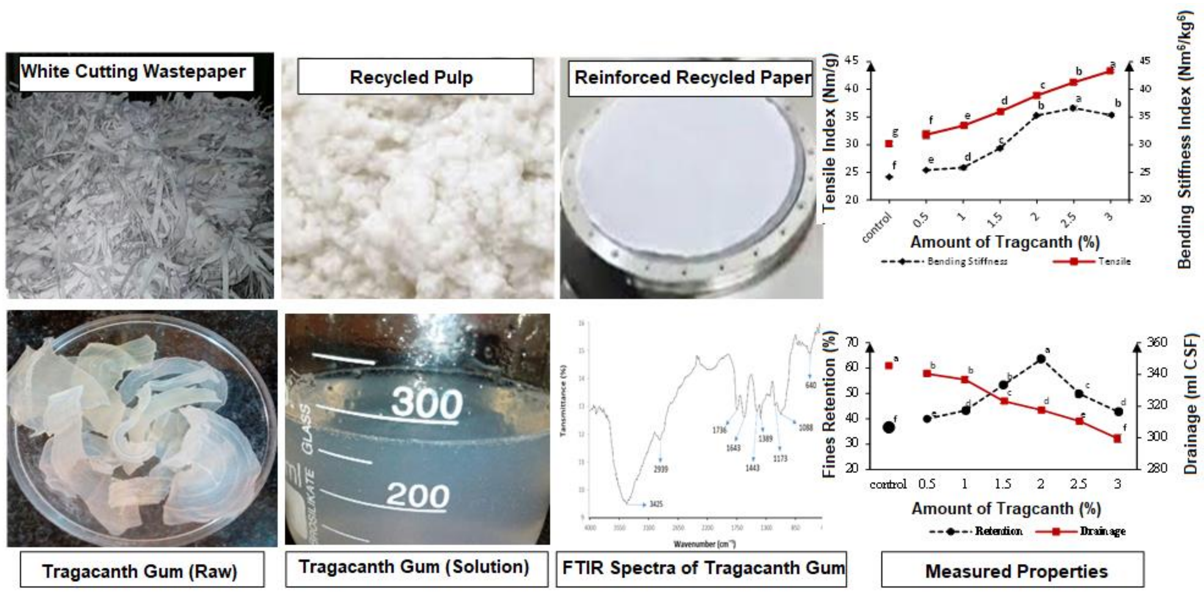
GRAPHICAL ABSTRACT



Upgrading Recycled Paper Using *Astragalus gossypinus* Tragacanth Gum as a Bio-based Additive

Mehdi Rahmaninia,[a,]* Yasin Rahmati,[a] and Mehdi Tabarsa [b]

Using environmentally friendly additives has been considered widely in different industries, especially papermaking, which has a high dependency on additives. The current study focused on applying a plant-based gum obtained from *Astragalus gossypinus* (a well-known plant in some regions of the world, especially Iran) in the papermaking process. The gum characterization showed a high content (about 84%) of carbohydrates (mainly hemicellulose with xyloarabinan monomers in the main chain and about 8% of uronic acid in the side chains), low ash content (2.58%) and insignificant protein content. FTIR spectra confirmed the structural results. The weight average molecular weight (M_w) and polydispersity of tragacanth gum were 4867 × 10^3 g/mol and 1.423, respectively. Considering the mechanical strengths results, applying the gum in recycled pulp improved tensile, burst, bending, and tear indices significantly. Moreover, fines retention experienced a significant increment by applying up to 2% of the gum. The pulp drainage decreased consequently by increasing the dosage of gum. The FESEM images confirmed the higher retention and bonding in paper structure by applying the gum. The results seem to open a new door for the application of different plant gums as a green additive for papermaking industries.

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Keywords: Recycled fibers; Green chemistry; Hemicellulose; Environmental concern

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INTRODUCTION

Papermaking has shown continued growth, but it has faced different challenges, especially the shortage of lignocellulosic raw materials (Rahmaninia and Khosravani 2015; Sangtarashani *et al.* 2020). Using the recycled lignocellulosic fibers has been considered as an important solution, and at least 50% of fibers used in papermaking comes from recycling mills (FAO 2021). But using recycled fibers can affect the properties of process and final product negatively. Papermakers have applied different ways for optimizing the recycled papers (such as mechanical treatment of fibers, mixing with virgin pulp, chemical and biological pretreatment of recycled fibers, applying different chemical additives in recycled pulp, *etc*.). Application of chemical additives for different purposes in paper recycling is a traditional way for improving the products and process quality (Sabazoodkhiz *et al.* 2017). Currently, the bio-additives have gained wide attention in industrial applications (Milani *et al.* 2024).

Hemicellulose is a biocomponent with significant amounts of hydroxyl and carboxyl groups in its structure. The presence of these active functional groups gives special characteristics to hemicelluloses, including good water absorption, which consequently can improve fibers swelling, flexibility, and bonding in paper structure. Moreover, this component shows a desirable solubility even in cold water making it a good candidate with easy and quick preparation in comparison with other additives (Lan *et al.* 2016).

Hemicelluloses are found in different plants; therefore, various plants could be used as a source of hemicellulose for different purposes. *Astragalus gossypinus* is a well-known plant, and its tragacanth gum has a huge amount of hemicellulose. Iran is one of the most important countries for producing tragacanth gum in the world, and about 500 tons of this gum has been exported in 2020. Tragacanth gum is a qualified hydrocolloid in the list of generally recognized as a safe (GRAS) ingredient of food and drug administration (FDA); it has applications in different industrial sectors (such as pharmaceutical, health and food industries) (Didar 2016). Structurally tragacanth gum is a highly branched, heterogonous, and hydrophilic carbohydrate with high molecular weight. This complex polysaccharide is odorless and slightly acidic. Tragacanth gum mostly consists of two polysaccharides, bassorin (tragacanthic acid) and tragacanthin, which are physically mixed with no chemical interaction. Bassorin, which constitutes 60 to 70% of tragacanth gum, is insoluble in water but can swell to form a gel. In contrast, tragacanthin is completely water soluble and forms a colloidal solution (Gavlighi *et al.* 2013).

Hemicellulose has been reported as an additive in papermaking (Basu *et al.* 2021). Using the stored hemicellulose in the seed of some plants (xyloglucan and galactomannan) in wet end chemistry of papermaking decreases the consumption energy and time of refining. Also, the mechanical properties of final papers are improved by the mentioned treatment without any reduction in optical properties (Lima *et al.* 2003). Applying xyloglucan to bleached softwood kraft pulp improves the formation and mechanical properties of produced papers (Christiernin *et al.* 2003). The addition of hemicellulose to bleached bagasse pulp could improve the mechanical and physical properties (Ads *et al.* 2015). Furthermore, the carboxymethylated hemicellulose isolated and modified from corncob increases the mechanical strength of paper (Zhao *et al.* 2018).

Although application of hemicellulose in papermaking has a long history, there continues to be a need for further research. Considering the tragacanth gum (as a rich source of polysaccharides especially hemicellulose) in papermaking process will be an interesting and new vision. These results will promote the application of different plant gums as green additives for the pulp and paper industries.

EXPERIMENTAL

White cutting wastepaper (printing and writing waste paper) was collected locally from Noor city, Iran. The commercial tragacanth gum of *Astragalus gossypinus* was prepared from Isfahan Province, Iran. This kind of tragacanth gum had a proper quality (white color with no impurity) (Fig. 1).

Preparation of Tragacanth Gum

The tragacanth gum initially was ground and dissolved in 25 °C distilled water with magnet stirrer (1000 rpm for 20 min) for preparation of solution with 0.5% concentration (Fig. 1).

Fig. 1. Plate form tragacanth gum (Left), tragacanth gum powder (Middele), tragacanth gum soultion in disstiled water (Right)

Analysis of Tragacanth Gum

Monosaccharide composition of tragacanth gum

For determination of monosaccharide composition of tragacanth gum, this polysaccharide was hydrolyzed with 4 M trifluoroacetic acid (TFA) in 100 °C for 6 h. The reduction and acetylation processes of monosaccharides was done using sodium borodeuteride (NaBD4) and acetic acid (Ciucanu and Kerek 1984). The produced derivatives were analyzed by Gas Chromatography Mass Spectrometer equipped with HP-5MS column (Agilent Technologies, CA, USA) (Tabarsa *et al.* 2017).

Determination of average molecular weight

First, 2 mg/mL polysaccharide was dissolved in distilled water with heating in microwave for 30 s. The prepared solution was filtered through a 3.0-μm cellulose membrane (Whatman International) immediately. A multi-angle static laser light scattering system (Technology Corp, Santa Barbara, CA, USA HELEOS; Wyatt) coupled with refractive index detection system (HPSEC-UV-MALLS-RI) and TSK G5000PW column (7.5 × 600 mm; Toso Biosep, Montgomeryville, PA, USA) was used for determination of the molecular weight characteristics. The mobile phase consisting of 0.15 M $NaNO_3$ and 0.02% NaN_3 was used at a flow rate of 0.4 mm/min. ASTRA 3.5 (Wyatt Technology Corp) software was employed to calculate the average molecular weight (M_w) and radius of gyration (R_g) (Wyatt Technology Corp.). The specific volume for gyration (SV_g) was calculated by the following equation (You and Lim 2000),

$$SV_g = 4/3\pi\ (R_g \times 108)^3/(M_w/N) = 2.522\ R_g^3/M_w \qquad (1)$$

where N is Avogadro's number (6.02 ×10^{23}/mol) and M_w, R_g, and SV_g units are g/mol, nm, and cm^3/g, respectively.

Determination of protein content, uronic acid content, and neutral sugar

The protein content was determined according to the method suggested by Lowry *et al*. (1951). The neutral sugars (sugars with no carbonyl and carboxyl groups) of tragacanth gum were measured according to phenol-sulfuric acid method (Dubois *et al*. 1956). Uronic acids of tragacanth gum were determined as suggested by Filisetti-Cozzi and Carpita (1991).

Fourier transform infrared (FTIR) analysis

Functional groups of gum hemicelluloses were analyzed by FTIR spectrometer (Shimadzu 8400s, Japan) in the range of 400 to 4000 cm^{-1}.

Preparation of Pulp Suspension

The White cutting wastepaper (WCW) reached ambient condition in paper recycling lab of Tarbiat Modares University after 24 h. Then the WCW was soaked in deionized water for a night and repulped and refined with valley beater up to 350 mL CSF according to TAPPI T200 sp-01 (2001). The prepared pulp was stored in a cold room for next steps.

Analysis of WCW

Ash content

Ash content of WCW was measured according to TAPPI T211 om-07 (2007) in 525± 25 °C for 4 h with an electric furnace.

Fines content

The fines content of recycled WCW pulp was determined according to TAPPI T261 cm-00 (2000) with a dynamic drainage jar (DDJ).

Papermaking process analysis

Fines retention

The fillers/fiber fines retention was determined according TAPPI standard T261 cm-00 (2000). In this respect, the dynamic drainage jar (DDJ) was used.

Pulp drainage

The pulp drainage was done according TAPPI standard T227 om- 04 (2004) with Canadian standard freeness (CSF) tester.

Preparation of handsheets

The paper sheets with 60 g/m^2 basis weight were produced with mold hand-sheet maker according to TAPPI standard T205 sp-02 (2002).

Characterization of Paper Sheets

The determined physical/mechanical properties of produced papers and the related TAPPI test methods are listed in Table 1.

Table 1. List of Determined Physical/Mechanical Properties of Papers

Characteristics	Measurements	Standards
Physical	Ash (%)	TAPPI T211 om-07 (2007)
	Thickness (mm)	TAPPI T411 om-05 (2005)
	grammage (g/m^2)	TAPPI T410 om-02 (2002)
	Elastic modulus (MP) / Tensile index (Nm/g)	TAPPI T494 om-01 (2001)
	Tear index ($mN.m^2/g$)	TAPPI T414 om-04 (2004)
	Burst index ($kPa.m^2/g$)	TAPPI T403 om-97 (1997)
	Bending stiffness index (Nm^6/Kg^3)	TAPPI T489 om-04 (2004)

Field emission scanning electron microscopy (FE-SEM)

The FE-SEM technique (TESCAN MIRA3, Czechia Republic) was used to characterize the morphology of the produced handsheets.

Statistical Analysis

For determination of the properties, five replications were used. Completely randomized design (CRD) was applied for statistical analysis and Duncan's multiple range test (DMRT) for categorizing the averages. The Duncan grouping was alphabetically ascribed. The data with the same alphabetical grouping (same letter) didn't have significant statistical difference (5% error level). SPSS software (IBM, version 16.0, Armonk, USA) was used for statistical analysis.

RESULTS AND DISCUSSION

Recycled Pulp Characterization

Table 2 shows the WCW recycled pulp characteristics. Considering the ash content and fines content, it can be concluded that about 8% of fines content was related to fiber fines created during repulping and refining process. The fiber fines content is an important factor in papermaking and has been considered widely even as a new source for producing other value-added products (Sangtarashani *et al.* 2020; Bagheri *et al.* 2021).

Table 2. Characteristics of Recycled Waste Office Pulp

Conductivity (µs/cm)	pH	Ash or filler content (%)	Freeness (mL CSF)	Fines content (%)
600-700	7 to 7.5	16.43	340	24.6

Characterization of Tragacanth Gum

Tables 3 and 4 show the chemical composition and molecular characteristics of tragacanth gum. Considering the results, the main chain of *Astragalus gossypinus* hemicellulose consisted of xyloarabinan monomers. No protein was found in the gum, and the ash content was 2.58%.

Table 5 shows the molecular properties of tragacanth gum. The weight average molecular weight (M_w) and the number average molecular weight (M_n) of tragacanth gum was 4.867×10^3 g/mol and 3.420×10^3 g/mol, respectively. Also, the polydispersity (M_w/M_n) as a factor shows the molecular weight distribution of polysaccharides and its molecular weight homogeneity, in the tragacanth gum was about 1.423.

Table 3. Characterization of Tragacanth Gum

Property	Moisture	Carbohydrate	Uronic acid	Protein	Ash
Present (%)	4.592	84.6	8.228	0	2.58

Table 4. Types and Percentage of Sugars Available in Tragacanth Gum

Monosaccharide	Arabinose	Xylose	Galactose	Rhamnose	Mannose	Glucose
Present (%)	44.055	39.36	6.895	3.555	3.53	2.59

Table 5. Molecular Weight of Tragacanth Gum and its Polydispersity

molecular weight (Dalton)	M_w (g/mol)	M_n (g/mol)	Pd
	4.867×10^3	3.420×10^3	1.42

M_n: Number average molecular weight; M_w: weight average molecular weight; Pd=M_w/M_n: Polydispersity

FTIR Spectra of Tragacanth Gum

Figure 2 shows the FT-IR spectra of tragacanth gum (*Astragalus gossypinus*). The results of FTIR analysis are shown in Table 6.

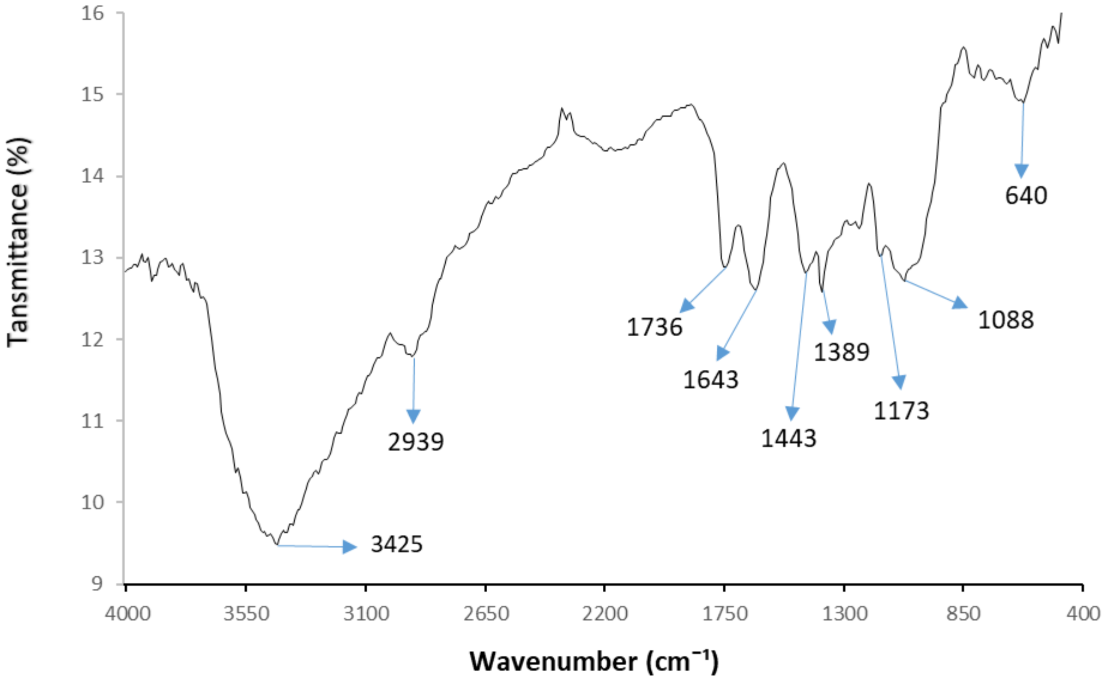

Fig. 2. FTIR spectra of tragacanth (*Astragalus gossypinus*)

Table 6. Assignment of FTIR Spectra of Gum Tragacanth (*Astragalus gossypinus*)

IR Band (cm^{-1})	Description	References
3425	stretching vibration of O–H groups	(Song and Hubbe 2014)
2939	C-H, O-H stretching vibration	(Sun *et al.* 2005)
1736	COOH ester groups	(Ahmad *et al.* 2010; Sun et al. 2005)
1643	H_2O, O-H	(Olsson and Salmén 2004; Sun *et al.* 2005)
1443, 1389	bending vibrations C-H, O-H groups	(Song and Hubbe 2014; Sun *et al.* 2005)
1173, 1088	C–O–C stretching of the glycosidic linkage between tragacanth monomers	(Song and Hubbe 2014; Sun *et al.* 2005)
640	H_2O, O-H	(Olsson and Salmén 2004)

Mechanical Properties of Produced Paper Sheets

Tensile and bending stiffness index

Tensile index is one of the important properties in papermaking. It is mainly related to bonding ability of different components in the paper sheet structure (*i.e.* fibers, fines, fillers, additives, *etc*.), sheet formation and the inherent fibers strength. Also, bending stiffness index, as an important property in different applications especially converting processes (such as printing), is mainly related to module of elasticity and the third power of thickness (Ek *et al.* 2009; Taheri *et al.* 2022).

Figure 3 shows the effect of tragacanth gum (*Astragalus gossypinus*) on the tensile index and bending stiffness index of recycled pulp. According to the results, the treated papers gained higher tensile and bending resistance compared to control papers (without gum treatment). Moreover, higher gum consumption positively affected the tensile and bending stiffness indices.

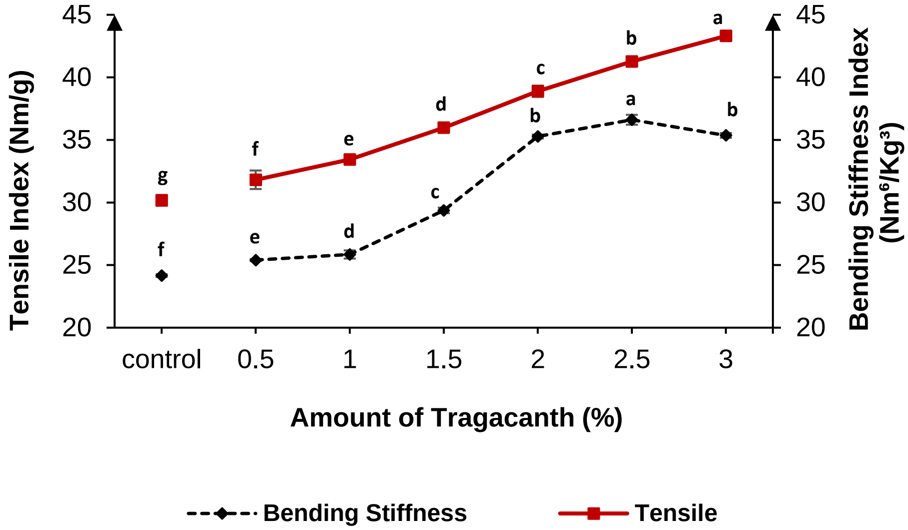

Fig. 3. The effect of tragacanth gum on the tensile index and bending stiffness index of recycled pulp. Plotted points that do not share the same letter were significantly different from each other (95% confidence level).

Tragacanth gum, with the main branch of xyloarabinan (containing a lot of hydroxyl groups) and also the presence of carboxyl groups related to uronic acid in side chains, had a significant effect on developing bonding in the recycled papers and so increasing the mentioned mechanical properties (Li *et al.* 2011). Lima *et al.* (2003) indicated that an unmodified polysaccharide such as hemicelluloses can increase the bonding of components (especially fibers and fiber fines) in the pulp suspension with different mechanisms such as electrostatic forces, hydrogen bonding, Van der Waals forces, and ionic bonding, which consequently would increase the tensile strength in produced paper sheets. Considering Table 7 (Didar 2016), it can be concluded that the increment of bending stiffness (as the same of tensile strength) is directly related to improving the bonding in recycled paper sheets and increasing in elastic module. In this respect, no significant changes were observed in paper thickness (Table 7).

Table 7. The Effect of Tragacanth Gum on Physical Properties of Recycled Papers

Treatment	Control	0.5	1	1.5	2	2.5	3
Thickness (mm)	0.1283	0.128	0.1284	0.128	0.128	0.128	0.128
Grammage (g/m^2)	58.2	58.59	60	60.5	60.9	61.4	60.9
Density (g/cm^3)	0.453	0.457	0.467	0.472	0.476	0.479	0.475
Elastic module(MPa)	1544	1809	2044	2358	2964	3412	2819

Burst and tear index

Tear strength is one of the important properties that can affect the papermaking process and paper converting process, especially in paper runnability. This property is mainly related to fiber length, inherent fiber strength, and fiber bonding. However it should be noted that while increment of interfiber bonds generally improves the paper strength, there's a threshold beyond which increasing the bonding may be detrimental to tear strength. Additional bonding can cause the papersheet to be brittle and cause fibers to break rather than pull out during the stressing, reducing the energy absorption capacity of the paper. Therefore, optimizing the degree of bonding is crucial especially when using strengthening additives. Also, the burst strength, as the same of tensile strength, is related to the state of bonding between paper components (fibers, fines, fillers, *etc*.) (Ek *et al.* 2009; Amiri *et al.* 2013).

Considering Fig. 4, the application of tragacanth gum in the recycled pulp had positive effect on the tear and burst indices. In both tear and burst strengths, an increasing trend can be observed by applying higher dosages of gum (up to 3% based on the oven dried pulp). In tear index, as two main factors, *i.e.*, the fiber length and inherent fiber strength, don’t have any changes (because of the constant fiber source), so logical and proportionate improvement in bonding between fibers in the structure of produced papers with applying the gum can be the main reason for the indicated results. As mentioned, applying the gum (with high hydroxyl and carboxyl groups) up to 3% (based oven dry pulp) (Tables 3, 4, and 6) can increase the bonding potential among the pulp suspension components (Li *et al.* 2011; Ads *et al.* 2015) without making the papersheet structure to be brittle.

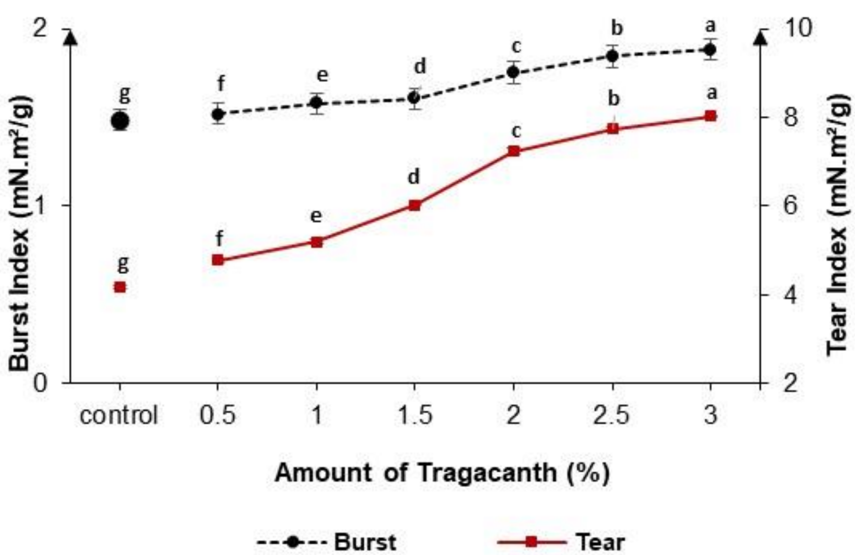

Fig. 4. The effect of Tragacanth gum on the burst and tear indices of recycled pulp. Plotted points that do not share the same letter were significantly different from each other (95% confidence level).

Process Properties

Fines retention and drainage

Fines retention and drainage are the main process properties which can affect other process and product properties (Sabazoodkhiz *et al.* 2017). Drainage is a critical factor in producing high basis weight paper products, especially affecting the productivity, energy costs in pressing and drying process, paper runnability and production efficiency (Kermanian *et al.* 2019). Both retention and drainage can be improved with different procedures, especially internal application of different additives (Gal *et al.* 2023). These additives usually impress these factors with fiber-fiber and fiber-fine flocculation mechanism (Rahmaninia *et al.* 2018).

According to Fig. 5, application of tragacanth gum in the recycled pulp improved the fines retention. It seems that the chemical structure of gum, with xyloarabinan monomers in the main chain and uronic acid groups in side chains, provides a lot of hydroxyl and carboxyl groups which play an important role in pulp flocculation with different mechanism, especially bridging among pulp components using electrostatic forces, hydrogen bonding, Van der Waals forces, ionic bonding (Rojas and Neuman 1999; Lima *et al.* 2003). The result of ash content (Table 8) confirmed the positive effect of gum on filler retention in recycled pulp.

Also, considering the results, addition of gum to recycled pulp decreased the drainage. Tragacanth gum (containing hemicelluloses full of hydroxyl and carboxyl groups) has a hydrophilic behavior (Balaghi *et al.* 2010; Didar 2016), and therefore, its application in pulp would decrease the drainage.

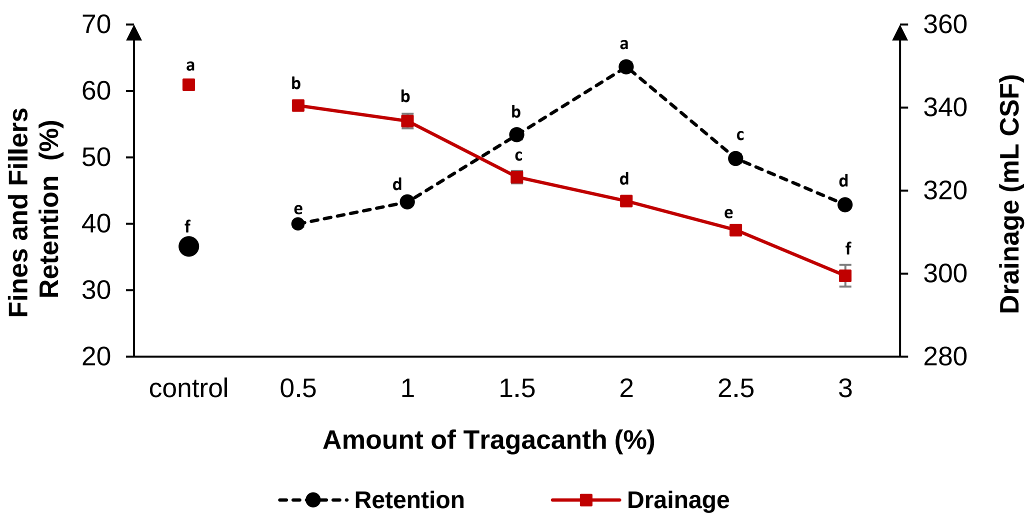

Fig. 5. The effect of tragacanth gum on fine retention and drainage of recycled pulp. Plotted points that do not share the same letter were significantly different from each other (95% confidence level).

Table 8. The Effect of Tragacanth Gum on the Ash Content of Recycled Papers

Treatment (%)	Control	0.5	1	1.5	2	2.5	3
Ash(%)	9.27	10.16	10.76	11.5	11.71	11.29	11.12

FE-SEM images of handsheets

Figure 6 shows the FE-SEM images of recycled paper sheets surface in control samples (no gum treatment) (Fig. 6a and b) and papers containing 2% gum (Fig. 6b and c).

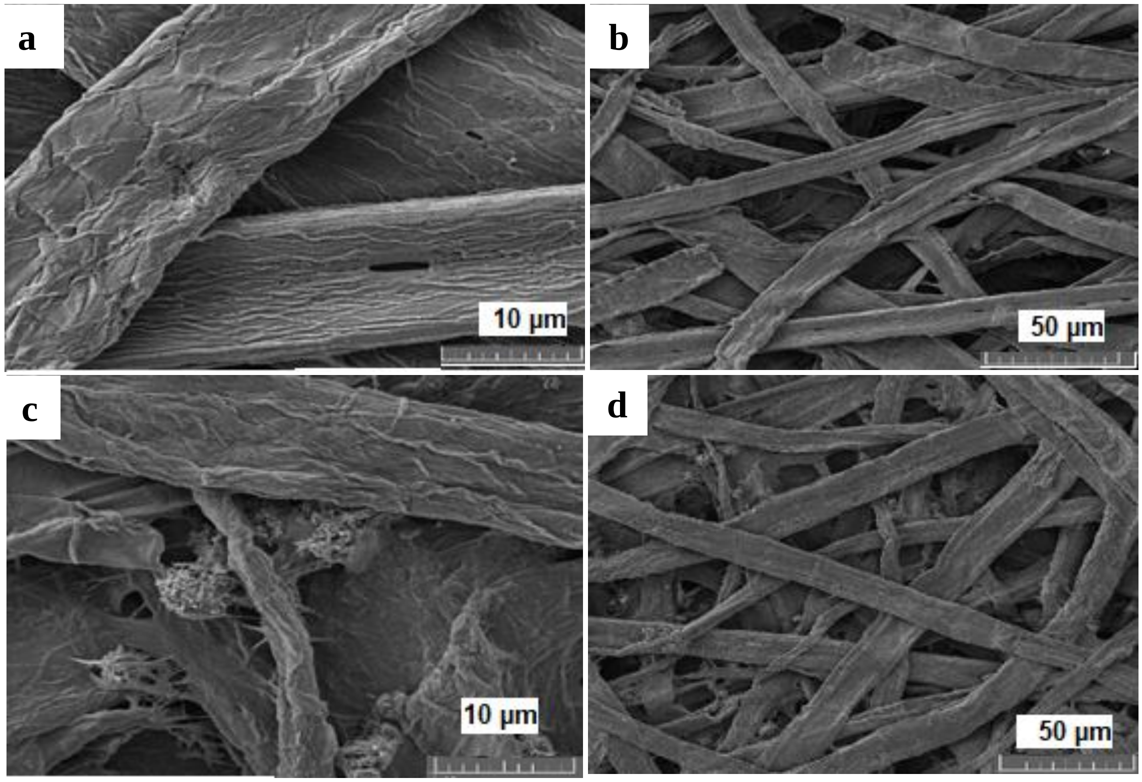

Fig. 6. FE-SEM images of recycled paper sheets surface in control samples (no gum treatment) (a and b) and papers containing 2% gum (c and d)

The surface of papers treated with tragacanth gum exhibited a denser structure compared to control sample that shows open structure with less bonding among the components. In Fig. 6c, the flocculated fillers can be seen clearly associated to fibers, which confirms the role of gum as a successful retention aid mentioned in the previous section.

CONCLUSIONS

1. The current research considered the possibility of using plant gums as a green bio-additive in cellulosic industry, especially the environmentally friendly recycling process. The tragacanth gum of *Astragalus gossypinus* as an abundant plant in some regions of the world, especially Iran, was considered as a novel candidate.
2. Tragacanth gum consists of high carbohydrate (hemicellulose) content (about 84%), ash content (about 2.58%), uronic acid content (8.23), insignificant protein content, and remaining amount as moisture content.
3. Moreover, the tragacanth gum hemicellulose was analyzed and the results showed that its monomeric units consist of arabinose (≈ 44.1%), xylose (≈ 39.3%), galactose (≈ 6.9%), rhamnose (≈ 3.6%), mannose (≈3.5%), and glucose (≈ 2.6%).
4. Considering the results, the main chain of *Astragalus gossypinus* hemicellulose consisted of xyloarabinan monomers in the main chain and uronic acid in the side chains.

5. The FT-IR spectra confirmed the mentioned results. Furthermore, the weight average molecular weight and the number average molecular weight of tragacanth gum was 4.867 × 103 g/mol and 3.420 × 103 g/mol, respectively with 1.42 polydispersity.
6. Also, the mechanical results of gum application as an internal additive in recycled paper showed significant improvement of tensile, burst, bending, and tear indices. Increasing the gum dosages has a positive effect on mechanical properties.
7. Although the drainage experienced reduction by applying the gum, the fines retention increased. The field emission scanning electron microscopy (FE-SEM) images confirmed the higher retention and bonding in paper structure by applying the gum.
8. The current research showed the favorability of tragacanth gum as an effective green bio-additive for internal application of papermaking. It can be regarded as an example of a new class of green additives for this industry.

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