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Insights into Gum Arabic interactions with cellulose: Strengthening effects on tissue paper

A. Cláudia S. Ferreira [a], Roberto Aguado [a,b,*], Ana M.M.S. Carta [c], Raquel Bértolo [c], Dina Murtinho [a], Artur J.M. Valente [a,**]

[a] University of Coimbra, CQC, Department of Chemistry, Rua Larga, Coimbra 3004-535, Portugal
[b] LEPAMAP-PRODIS Research Group, University of Girona, M Aurèlia Capmany, n°61, Girona 17003, Spain
[c] RAIZ - Forest and Paper Research Institute, Quinta de S. Francisco, Apartado 15, Eixo-Aveiro 3801-501, Portugal

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ABSTRACT

Competitiveness in the market for tissue paper, the only paper grade whose consumption increased during 2020 in Europe, requires seeking viable options to continuously improve its properties. This work explores the combination of gum Arabic, which is a naturally found, biodegradable, cheap and versatile heteropolymer, with bleached cellulosic pulps. Blends of pulps and alkaline gum Arabic (GA_b) were analyzed in terms of their thermal degradation behavior, morphology, water absorptivity and drying rate. It was found that water uptake increased by 23% for the maximum proportion of GA_b tested (30%, w/w) and that water desorption followed quasi-zero-order kinetics. Furthermore, these blends were used to prepare light-weight paper handsheets by bulk addition, in which small proportions of GA_b (3%) were enough to significantly improve tensile strength. Remarkably, the most unexpected result came from the surface addition of alkaline GA_b by spray coating, as the tensile index was increased from 7 to 18 N m g^{-1}. Overall, even though it was not possible to improve and enhance simultaneously all properties, the use of pre-blended GA_b in papermaking revealed significant potential when it comes to increasing strength, a key property in different tissue products, such as multipurpose and kitchen rolls, or even in handtowels.

1. Introduction

Tissue paper, one of the most popular paper grades, is characterized by its low basis weight, high absorbency, low density and, at least when intended for hygienic purposes, softness [1,2]. It is a well-established industry, encompassing essential daily commodities such as facial tissue, paper towels, toilet paper, and napkins [3,4]· This industry, unlike that of printing paper, is growing even in European countries. While the European consumption of graphic paper decreased by 18% along 2020, partly because of the Covid-19 crisis and by its replacement with digital communication, paper for sanitary and household consumption has shown an increase of about 4% [5]. The conditions are right for greater competitiveness in these markets, which means a constant looking for alternatives to reduce costs and enhance the tissue paper performance. At the same time, and not less importantly, such enhancement should be attained by eco-friendly, "green" means [2,6,7]·

In general, the key properties of tissue paper are water absorption capacity, softness and tensile strength [1,8]. Their relative importance, and thus the reason why a costumer will choose a certain brand over another one, depends on the specific application of the end product [9, 10]. For instance, in the case of kitchen towels, consumers usually expect a strong product even in wet conditions, with high absorbency. Likewise, bath tissue needs to be strong enough when wet, but being as soft as possible is also desired. These key properties are influenced by the manufacturing process, the origin and the type of fibers, and the chemical and mechanical treatments that they undergo [11,12].

Framed in a research line of different additives to enhance some properties of tissue papers, including nanofibers and essential oils [4, 13], we explore the possibilities of pre-blending a part of the pulp with gum Arabic. Gum Arabic is an amorphous, non-toxic, odorless, colorless and tasteless natural polysaccharide [14–16]. It has failed as a stabilizing agent for papermaking in the past [17], when directly added to the

Abbreviations: BEKP, Bleached eucalyptus kraft pulp; GAb, Alkalized gum Arabic; TS7, Real softness test; TS750, Smoothness test.

* Corresponding author at: LEPAMAP-PRODIS Research Group, University of Girona, M Aurèlia Capmany, n°61, Girona 17003, Spain.
** Corresponding author.
E-mail addresses: roberto.aguado@udg.edu (R. Aguado), avalente@ci.uc.pt (A.J.M. Valente).

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suspension of fibers and fillers, but the pre-blending approach is hypothesized to attain favorable interactions with cellulose. This approach takes into account that gum Arabic is water-soluble, amphiphilic, with good affinity towards both cellulose and water molecules, and their solutions can be highly concentrated without being excessively viscous.

In our work, gum Arabic was pre-treated in an alkaline solution (GA_b) to increase their water absorbency. Blends of pulp with different percentages of gum Arabic were prepared and characterized, studying their water uptake capacity and desorption kinetics. Afterwards, key tissue paper properties were evaluated by adding this carbohydrate to the pulp by two different methods: bulk addition (sheet forming) and surface addition (spray coating). Throughout facile and feasible methods of incorporation, we found great results in terms of paper strength.

2. Experimental section

2.1. Materials

Bleached eucalyptus kraft pulp (BEKP, short fiber, 35°SR) and bleached softwood pulp (long fiber) were kindly provided by a Portuguese tissue mill. Gum Arabic from acacia tree, a branched heteropolysaccharide with an average molecular weight of approximately 250,000 g mol^{-1}, was purchased from Sigma-Aldrich. Acetone and sodium hydroxide were acquired from LabChem and José Manuel Gomes Santos Lda., respectively.

The carboxyl content of gum Arabic, mainly provided by D-glucuronic acid, was measured by volumetric titration with NaOH, using phenolphthalein as indicator. It was found to be (128 ± 2) μmol –COOH g^{-1}.

2.2. Blending

First, gum Arabic was submitted to an alkaline treatment in order to deprotonate its carboxyl groups. This treatment consisted of the dissolution of this polymer in excess of a sodium hydroxide solution (0.5 M) and subsequent precipitation with acetone. The precipitated solid (GA_b) was dried in an oven set at 105 °C and stored in a vacuum desiccator until further use.

Samples of GA_b were dissolved in distilled water to different concentrations (1–30%, w/w). Then, BEKP was added to the various solutions in proportions of 9:1 or 7:3 to GA_b. The heterogeneous mixtures were stirred for 24 h at 23 °C, allowing for fiber swelling and the establishment of as much hydrogen bonding as possible. The suspensions were poured into poly(methyl pentane) containers and left to evaporate at 60 °C.

2.3. Characterization of GA_b-containing pulp blends

The thermogravimetric analysis (TGA) of samples was carried out with a TG209 F3 Tarsus thermogravimetric analyzer (Netzsch Instruments), at a heating rate of 10 °C min^{-1}, from 25 °C to 600 °C, under nitrogen atmosphere (flow rate of 20 mL min^{-1}).

Water absorption and water desorption kinetics were evaluated. For water uptake, blended samples with different formulations were immersed in water, at 25 °C, overnight. After that, the excess water was removed, and the samples were weighed. Samples were dried in an oven at 60 ºC for at least 24 h and then placed in a desiccator, for 4 h, to cool down. They were weighed again, and the percentage water uptake, H_P, was calculated using Eq. (1).

$$H_P(\%) = \frac{m_{swelling} - m_{dry}}{m_{dry}} \times 100 \quad (1)$$

where $m_{swelling}$ is the mass of each sample, in equilibrium with water, and m_{dry} is its mass after oven-drying.

Table 1
Handsheets formulations for in terms of mass of BEKP (short fiber), softwood pulp (long fiber), pulp+GA_b blend (modified fiber).

Formulations	Ref.	GA3%	GA7%	GA9%⊥
Long fiber (%)	30	30	30	30
Short fiber (%)	70	40	–	40
Blend* (%)	0	30	70	30

* Pulp + 10% GA_b (for GA3% and GA7%); pulp + 30% GA_b (for GA9%).

Table 2
Handsheets formulations for spray (GA_b solution) incorporations. The percentage of additive refers to the concentration (w/w) of the sprayed solution in each case.

Formulations	Ref.	GAb2%	GAb5%	GAb10%	GAb13.5%
Long fiber (%)	30	30	30	30	30
Short fiber (%)	70	70	70	70	70
Additive (%)	0	2	5	10	13.5

Table 3
Fitting parameters obtained from the desorption kinetics of water from different blends, by using Eq. (2).

	k (10^{-3} s^{-n})	n	R^2
Pulp	0.708 ± 0.01	0.961 ± 0.007	0.999
Pulp+ 1%GAb	1.09 ± 0.03	0.904 ± 0.009	0.997
Pulp+ 5%GAb	1.84 ± 0.02	0.858 ± 0.004	0.999
Pulp+ 10%GAb	2.02 ± 0.02	0.832 ± 0.004	0.999
Pulp+ 20%GAb	2.74 ± 0.02	0.824 ± 0.003	1.000
Pulp+ 30%GAb	5.65 ± 0.13	0.726 ± 0.010	0.997

For the kinetic studies the samples were treated the same way as in the water uptake studies. They were dried in a heating balance (A&D MS-70, equipped with a super hybrid sensor) that was set at 80 ºC. Then, the weight of each of the samples was recorded at different times, t, up to reach equilibrium. The kinetics of water desorption was fitted to an empirical power function, as in Eq. (2):

$$\frac{m_t}{m_{eq}} = kt^n \quad (2)$$

where m_t is the cumulative weight of water released at each time (t) and m_{eq} is the cumulative weight of water released at equilibrium.

In order to elucidate the surface morphology of blends, scanning electron microscopy (SEM) was used. A TM4000 Plus tabletop microscope from Hitachi provided backscattered-electron imaging, working at high acceleration voltage (15 kV) to attain an acceptable depth of field while keeping a good resolution at relatively low magnification levels (×800).

2.4. Tissue paper properties

Laboratorial sheets were produced with low basis weight (20 g/m^2), according to an adaptation of the ISO standard 5269-1. Gum Arabic was incorporated by two different means: in bulk, accounting for the three sets of samples that are summarized in Table 1, and spray coating, as listed in Tables 2 and 3. Since direct bulk addition would be unsuccessful in the absence of a cationic polyelectrolyte or crosslinker, this bulk incorporation was carried out with the aforementioned blends, along with unmodified pulp. For that, the blend and the pulp were briefly mixed under stirring, at low consistency (<1%), right before being poured into the sheet former. As for spraying, aqueous solutions of GA_b accounting for concentrations of 13.5% or lower, so that the cinematic viscosity was always below 10 cSt (even at very low shear rates), were used to load a domestic hand sprayer. The sprayer was operated over the sheets after drying, in the same way and by the same experimenter in all

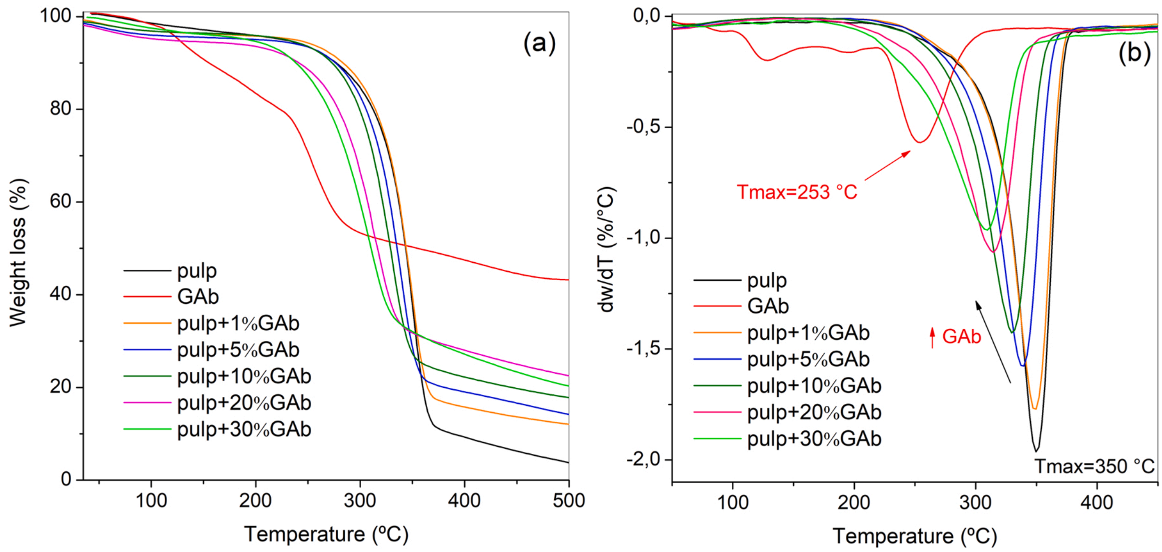

Fig. 1. Thermograms (a) and DTG curves (b) for the pulp+GA_b mixtures.

cases.

The handsheets obtained for each formulation were then pressed at 300 kPa for 5 min and dried under controlled conditions of temperature (23 ± 1 °C) and relative humidity (50 ± 2%). Subsequently, the paper properties that are most relevant for tissue grades were measured. Basis weight and bulk were determined following ISO 12625:6 and 12625:3, respectively, while tensile index was determined in a Vertical Tensile Tester (Frank-PTI) according to ISO 12625:4. Softness was analyzed on Tissue Softness Analyzer (TSA) from Emtec, a device comprising internal methods that estimate softness as the handfeel coefficient (HF), the so-called "real softness" (TS7) and "smoothness" (TS750). The algorithm used was QAI. The capillary rise was evaluated according to an adaptation of ISO 8787 on an Enrico Toniolo apparatus. On a Frank-PTI basket-immersion device, following an adaptation of ISO 12625–8, the water absorption capacity was determined. Air permeability was tested by means of FX3300 LabAir III equipment with a pressure drop of 200 Pa (FEXTEST Instruments).

2.5. Statistical considerations

Tolerance intervals were computed as twice the standard deviation around the average in each case. Since overlapping intervals do not necessarily imply that the difference between means could be deemed non-significant [18], one-way ANOVA tests were carried out to compare groups of datasets at two different GA_b levels. The significance level was defined as $\alpha = 0.05$. We can state the null hypothesis as follows: "the average of the differences between the sets of values that are being compared is zero". Then, the difference was deemed significant as long as the calculated p-value (p) was less than 0.05.

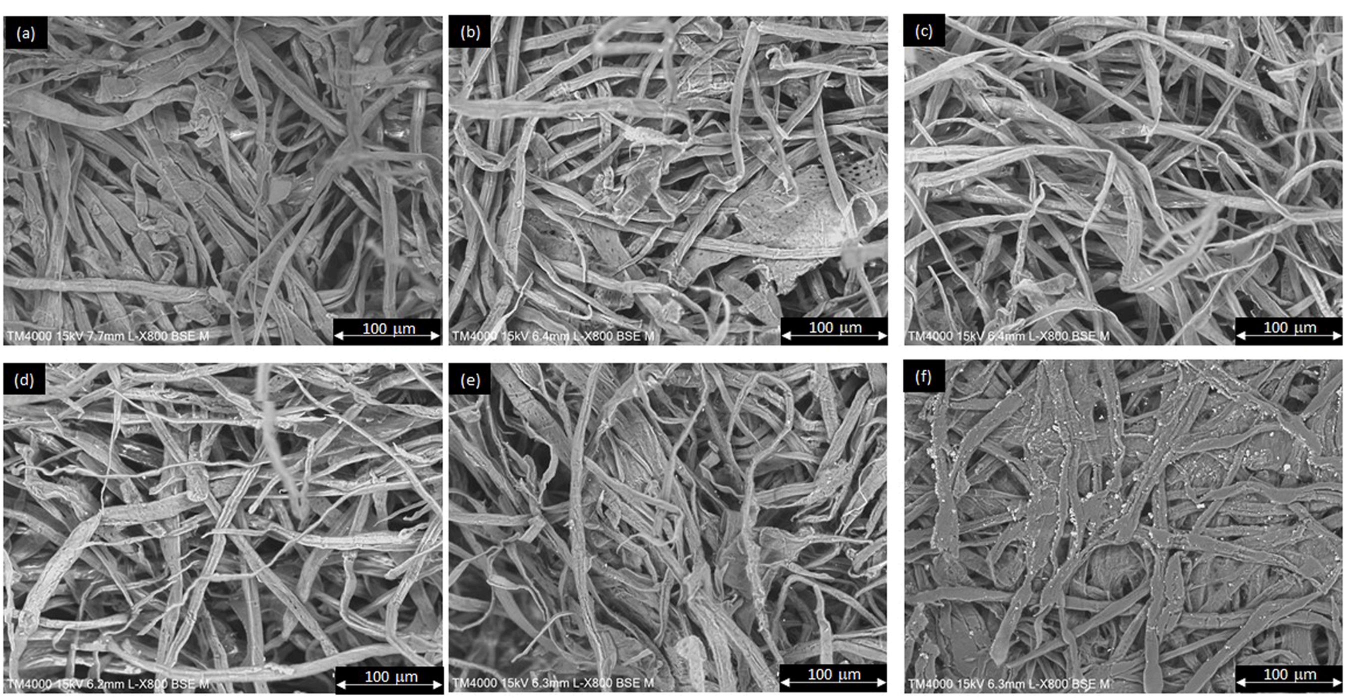

Fig. 2. SEM images of (a) pulp; (b) pulp + 1% GA_b; (c) pulp + 5% GA_b; (d) pulp + 10% GA_b; (e) pulp + 20% GA_b; (f) pulp + 30% GA_b.

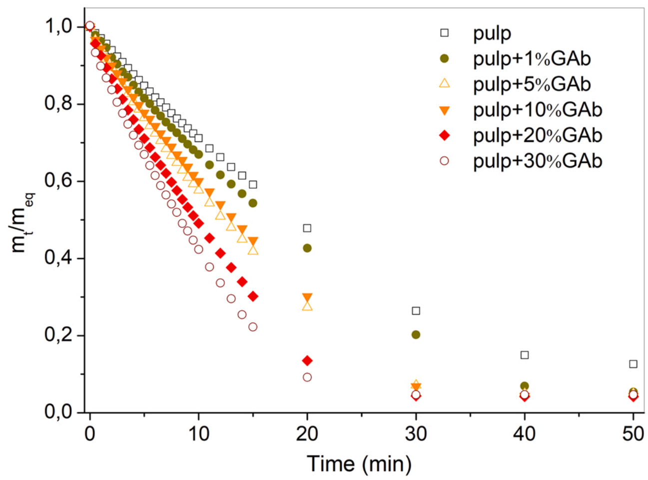

Fig. 3. Normalized water desorption kinetics from pulp and GA_b-containing pulp.

3. Results and discussion

3.1. Characterization of pulp-GA_b blends

The thermograms presented in Fig. 1a show that the thermal degradation of pulps is shifted towards lower temperatures with increasing GA_b content. Furthermore, the two characteristic transitions of GA_b following the loss of water by evaporation, a first one 130–195 °C and a second one whose T_{max} is 253 °C, cannot be distinguished in the TG curves for blends. Therefore, it is evident that blends of BEKP and GA_b, albeit non-covalent, are not random physical mixtures. Instead, the intermolecular forces established by mixing in an aqueous medium these polysaccharides, cellulose and GA_b, are presumably similar to those of cellulose-cellulose or cellulose-hemicellulose. These include hydrogen bonds between hydroxyl groups and hydrophobic interactions, among others [19,20].

In Fig. 2b, displaying the first derivative (DTG curves), we can observe that blending produced a decrease in the temperature of maximum weight loss rate (T_{max}) of pulp. More precisely, T_{max} goes from 350 °C (pulp) to 340 (5% GA_b), 330 (10% GA_b), 315 (20% GA_b), and 310 °C (30% GA_b). Overall, this reflects a plasticizing effect, which is consistently more notorious as the proportion of GA_b increases.

Micrographs highlighting the morphology of pulp+GA_b blends are presented in Fig. 2. Little difference is observed for the lowest proportions of GA_b, but the phenomenon of fibers becoming stuck together is more evident in the mixtures with 20% and 30%GA_b.

Therefore, gum Arabic seems to promote the densification and loss of porosity of the material. Not less importantly, and possibly owing to the amphiphilic nature of this heteropolymer, it does so by establishing strong intermolecular interactions, as inferred above from the thermal degradation behavior. This is highly relevant for sheet forming, as many other water-soluble additives that have the purpose of filling fiber-fiber spaces are easily lost through the wire [21,22].

The water desorption kinetics provides relevant information on the mechanism of interaction and, consequently, on the effect of GA_b on the blend. Fig. 3 shows the normalized water desorption from different blends. The desorption rate has been quantified by using a power law equation (Eq. 2), valid for short-range times, i.e. for $m_t/m_{eq} < 0.6$ [23]. The corresponding fitting parameters are summarized in Table 2. It can be concluded that for pulp, water desorption follows zero-order kinetics ($n \sim 1$), evidencing constant rate. However, by increasing the amount of GA_b incorporated in blends, n decreases, indicating that water diffused out by an anomalous (non-Fickian) behavior, which is indicative of coupling diffusional and relaxational mechanisms. As it will be discussed later, the change might be due to the GA plasticizer effect [24].

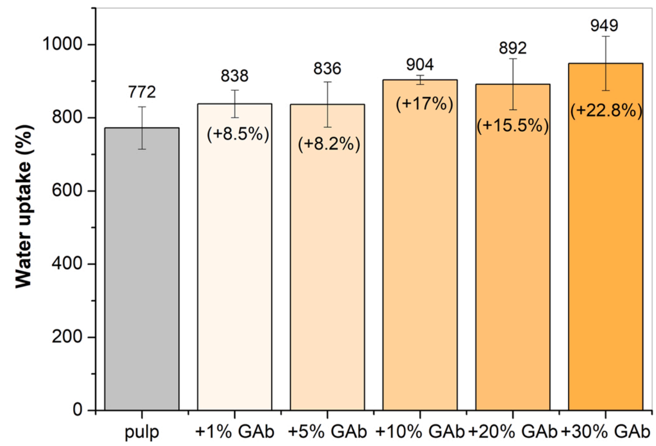

Fig. 4. Results of water uptake obtained for the different mixtures.

The results obtained in water uptake tests for the different mixtures of pulp with GA_b are shown in Fig. 4. As intended, water absorptivity, one of the key properties of tissue paper, was enhanced with increasing the fraction of GA_b, but the minimum percentage to attain a significant difference when compared to the control pulp was 10% ($p = 0.006$). When this fraction was 30%, the enhancement of water absorption capability was as high as +23%. Indeed, the highly hydrophilic groups of GA_b, particularly its carboxilates (–COO^- Na^+), favoured the interaction with water molecules. But it is worth stressing that, while virtually any carboxylated polymer would have fulfilled this requirement, the polysaccharide backbone of GA_b makes it stand out because of its capabilities of hydrogen bonding with cellulose.

3.2. Evaluation of key tissue paper properties

3.2.1. Bulk addition of gum Arabic

Figs. 5 and 6 show the results of important tests in papermaking, particularly for tissue paper grades, that were performed on laboratory sheets resulting from the bulk incorporation of pulp+GA_b blends (Table 1). Therefore, GA_b was allowed to interact with cellulose and hemicellulose in aqueous media twice. The first time was the preparation of the pulp+GA_b blend, in a similar way to the pre-reinforcement of a part of the pulp that has been yielded promising results in previous works [25,26]. The second step, requiring a much shorter time (due to the needs of the paper industry), was the dilution of this reinforced batch along with both short fiber and long fiber pulps.

In Fig. 5a, we observe a similar increase in tensile strength in the cases of low GA_b proportions (3% and 7%; $p \leq 0.004$) but it seems to decrease back when the weight percentage of GA_b is made up to 9%. For air permeability (Fig. 5b), taking into account the results of ANOVA tests, the addition of pre-blended GA_b (9%) exerted a significant decrease ($p = 0.019$). No significant difference ($p \geq 0.21$), however, was appreciated in what pertains to the water absorption capability results (Fig. 5c). Even from some due caution (given the large random errors), it can be concluded that the influence of bulk-added GA_b on air permeability is either null or negative, i.e., either decreasing or not affecting significantly the matrix porosity.

Klemm capillarity, represented in Fig. 6a, sees an increase with the progressive incorporation of alkaline gum Arabic. The formulation GA9% presents the highest values for this property. A further assessment has been done by using a first-order kinetics law equation, which integration leads to Eq. (3) [27]:

$$H = H_e - (H_e - H_0) \quad e^{-\frac{t}{t_c}} \tag{3}$$

where H_0 and H_e are a constant and equilibrium height, respectively, and t_c indicates the time required for water waterfront to reach $2/3H_e$.

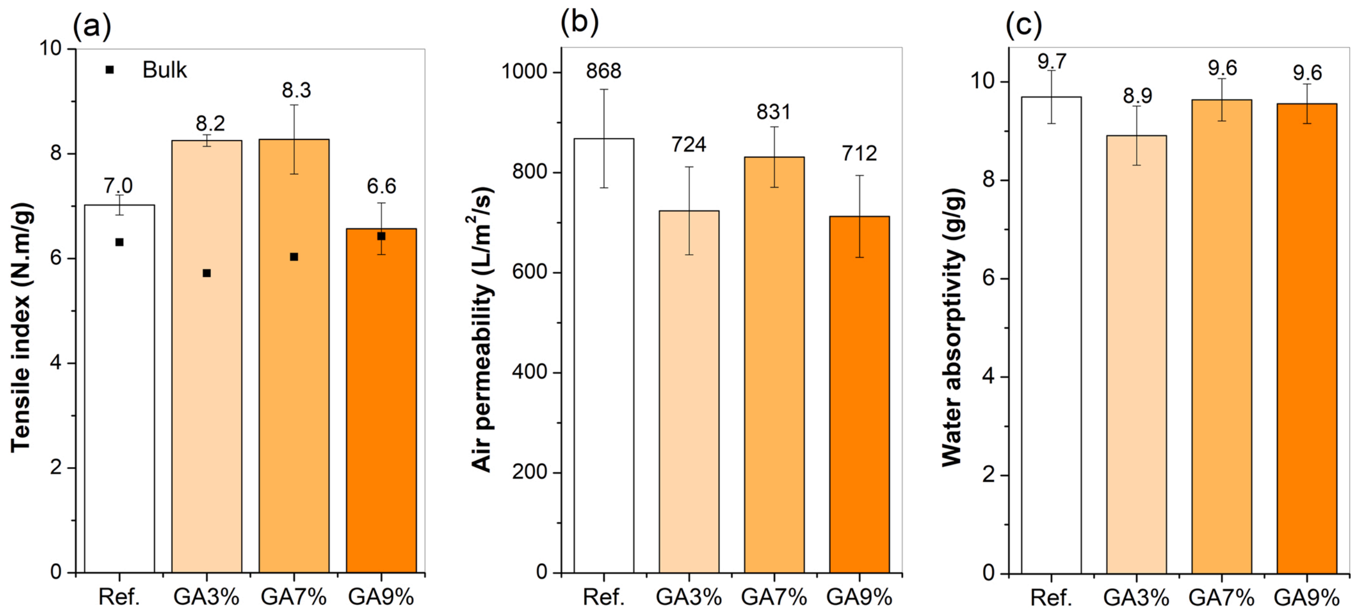

Fig. 5. Results of tensile index (a), air permeability (b), and water absorption capability (c) for the pulp+GA tissue handsheets of Table 1 (bulk addition).

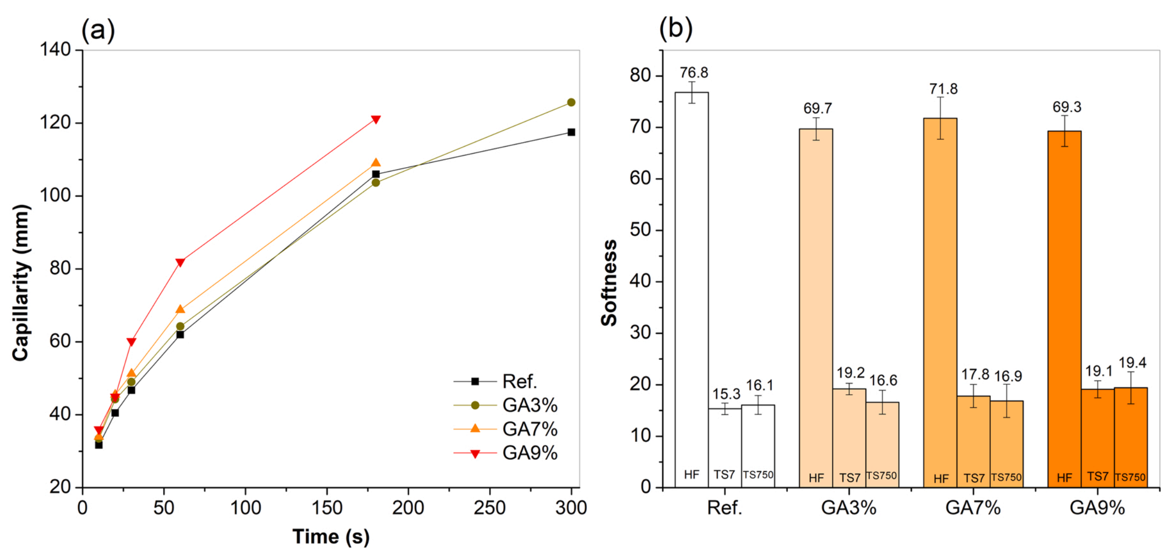

Fig. 6. Capillarity (a) and (b) softness for Table 1 tissue handsheets (bulk addition).

Table 4
Fitting parameters obtained from the capillary kinetics in paper samples that include GA_b, added either in bulk or on the surface, by using Eq. (3).

		H_0 (mm)	H_e (mm)	t_c (s)	R^2
Bulk addition	Ref. (paper)	24 ± 2	127 ± 4	120 ± 14	0.997
	Paper + 3%GA_b	29 ± 2	144 ± 7	168 ± 25	0.997
	Paper + 7%GA_b	27 ± 2	129 ± 8	110 ± 18	0.996
	Paper + 9%GA_b	21 ± 3	130 ± 5	72 ± 10	0.995
Surface addition	Paper + 2%GA_b	24 ± 4	120 ± 10	133 ± 4	0.998
	Paper + 5%GA_b	24 ± 1	123 ± 4	153 ± 2	0.998
	Paper + 10%GA_b	25 ± 2	143 ± 7	270 ± 4	0.993
	Paper + 13.5% GA_b	29 ± 3	133 ± 9	270 ± 6	0.989

From the analysis of the fitting parameters (Table 4), it can be seen that, taking into account the experimental error, H_e values do not depend on the presence of GA_b or the kind of addition ($p = 0.91$, ANOVA test). On the other hand, an increase in the amount of GA_b sprayed leads to an increase of the constant time t_c, except in the case of increasing the concentration of sprayed GAb from 10% to 13.5% (completely overlapping intervals).

Softness quantification is a tad more controversial, since there are different ways, all of them imperfect, of translating a sample measurement in a laboratory into a potential human perception [28]. That said, there is little room for doubt when all of these ways converge towards a similar conclusion. Fig. 6b displays the results for the three softness parameters analyzed: HF coefficient, TS7 and TS750. For handfeel, the alkaline gum Arabic had a negative effect. Consistently, both TS7 and TS750 increase for this samples. A higher TS7 means that was more obstruction to the blades of the analyzer and less mobility of superficial fibers. Likewise, higher TS750 represents higher paper roughness. In other terms, addition of GA_b made paper slightly rougher, less suitable

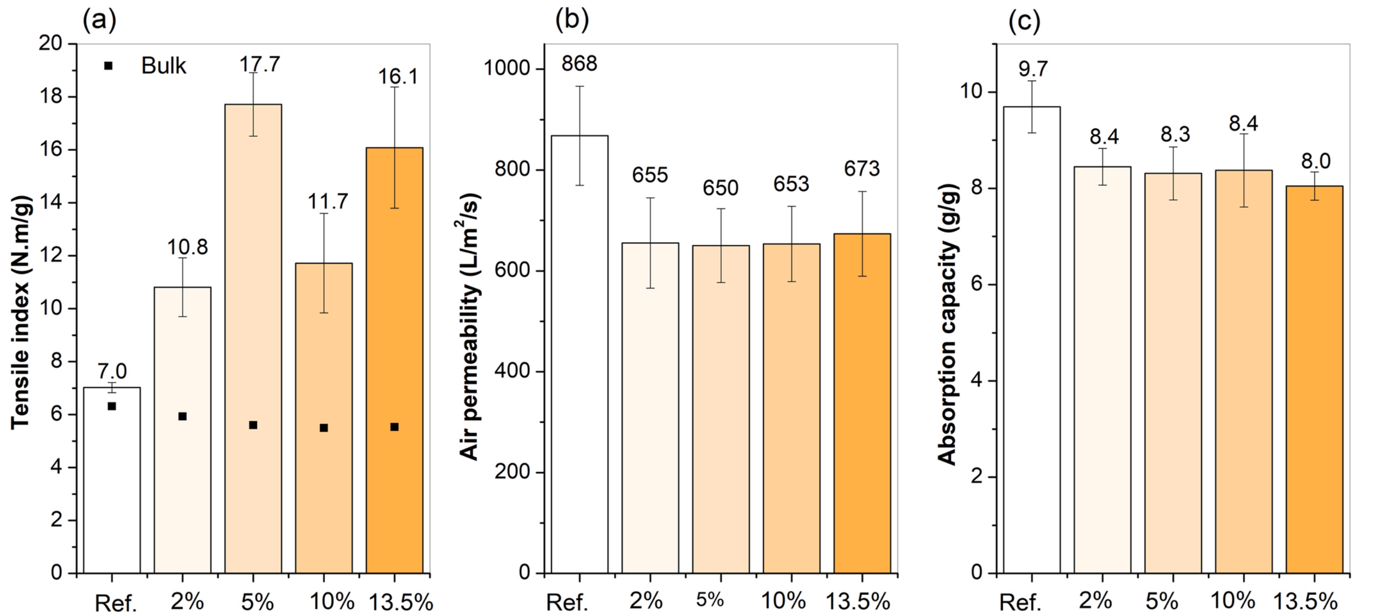

Fig. 7. Results of tensile index (a), air permeability (b), and water absorption capability (c) for the pulp+$_{GAb}$ tissue handsheets of Table 2 (spray coating).

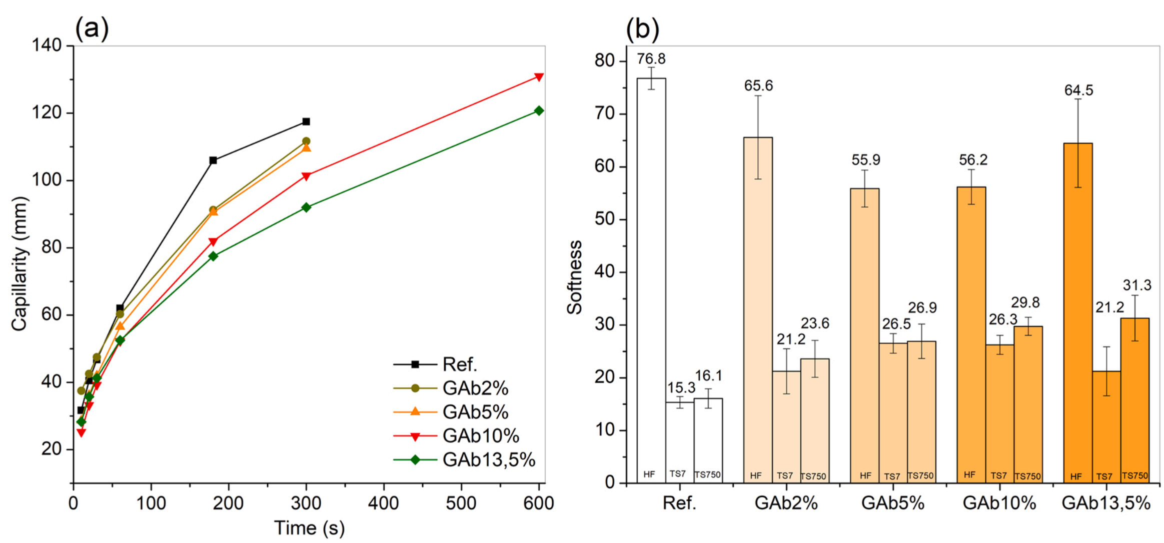

Fig. 8. Capillarity (a) and softness (b), expressed as different handfeel coefficients, for Table 2 tissue handsheets (spray coating).

for sanitary paper, but probably appropriate for cleaning purposes.

3.2.2. Surface addition of gum Arabic

The differences between the weight of each set of sheets before and after spraying, always at equilibrium moisture and under controlled conditions of temperature (23 °C, 50% relative humidity) revealed how much mass of GA_b was incorporated onto the surface of paper in each case. Concentrations equal to 2%, 5%, 10% and 13.5% resulted in weight gains of 1.13, 1.87, 2.70 and 5.15 g/m^2, respectively.

Figs. 7 and 8 present the results of key physical properties that were obtained for the handsheets coated by spraying the additive. This approach guarantees the additive presence in the final product, as virtually no loss of GA_b is possible. Furthermore, it takes place in only one step, not requiring the previous mixing step that bulk incorporation, due to the need to overcome electrostatic repulsion with hydrogen bonding, requires.

The enhancement of tensile index that can be seen from Fig. 7a was significant ($p = 5 \times 10^{-5}$ in the worst case). From the results obtained, we concluded that by using a 5% solution of alkaline gum Arabic, the tensile index had an increment of + 153%. Further additions, however, may promote fiber shrinkage upon drying.

Regarding air permeability (Fig. 7b) the spray application on the surface leads to a decrease of this property, i.e., air permeability is decreased ($p \leq 0.006$). The only setback for using GA_b by surface addition can be inferred from Fig. 7c, in which we observe that the absorption capacity decreases when compared with the reference handsheets ($p \leq 0.011$). Likely, a significant fraction of gum Arabic was quickly eluted in the paper absorptivity test. This was not possible when testing the water retention in pulp blends, where excess water was removed only by evaporation.

Capillarity (Fig. 8a) decreased with the amount of additive. Consistently with the discussion above, alkaline gum Arabic seems to promote hydrogen bonding and tightening the fiber network, and this blocked the water rise, increasing the t_c in ca. 125% (Table 4).

In Fig. 8b, we represent all the softness parameters obtained. As in additive bulk incorporations, the handfeel decreased and both TS7 and

TS750 increased. All considered, surface addition leads to a paper product that seems more suitable for food packaging (e.g., fluorine-free wraps) than for cleaning or sanitary uses.

4. Conclusions

Pulp+GA_b blends were successfully formed, taking advantage of the non-covalent interactions between the polysaccharide backbone of cellulose and GA. Thermogravimetric analysis of mixtures showed lower degradation temperatures, indicating a plasticizing effect. SEM analysis suggests that GA_b occupies the free spaces between the fibers, aggregating them, and thus acting as binder. Fitting the water desorption kinetics to a power function allowed us to suggest an anomalous mechanism. The water uptake of the blends increased with the amount of GA_b. Finally, papermaking results showed that the addition of this carbohydrate heteropolymer, either as bulk additive or as surface additive, increased the resistance of the paper, this being more accentuated when this application was performed by spray coating, by which the tensile index was increased by up to +153%.

In all cases, despite the losses on softness and absorption, the end product was deemed suitable for products such multipurpose and kitchen tissue rolls, or even handtowels, where strength is key relatively to the other properties (softness or absorption). Nonetheless, the bulk incorporation approach, with its enhanced capillarity, could be used towards high absorbent tissue kitchen rolls for water absorption optimization.

CRediT authorship contribution statement

A. Cláudia S. Ferreira: Methodology, Investigation, Writing – original draft. **Roberto Aguado**: Writing – original draft, Writing – review & editing, Visualization. **Ana M.M.S. Carta**: Methodology, Formal Analysis, Writing – review & editing. **Raquel Bértolo**: Investigation, Data curation, Validation. **Dina Murtinho**: Conceptualization, Validation, Resources. **Artur J.M. Valente**: Formal analysis, Supervision, Project administration, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Declarations of interest

none.

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