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Nano-processing of Apple Pomace for Cosmetics Ingredients

Audrey Zahra[1], Seo-Kyoung Lim[2], Hyun-A Seong[3‡] and Soo-Jeong Shin[4†]

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ABSTRACT

Apple is widely produced and consumed worldwide. Substantial amounts of unused apple by-products, such as pomace, are produced by industrial activities. This study investigated the rheological properties of apple pomace powder with different cultivars prepared using carboxymethylation to varying degrees of substitution (DS) up to DS 0.4 and nano-processing using a high-pressure homogenizer. We found a high carboxyl content on cellulose in apple pomace nanoparticle (APN) cultivars and enhanced water retention value as a humectant with increased DS during pretreatment. The addition of carboxymethylation also significantly increased the consistency and spreading ability of the APN hydrogel until optimal state and decreased it at higher DS. The APN hydrogel had better water retention value than viscose and alginate. It also had better consistency than commercial humectants (i.e., glycerol and collagen) and a spreading ability that was comparable to other thickening agents (i.e., Carbopol 940, 941, 980, and Aristoflex).

Keywords: *Apple pomace nanoparticles (APN), humectant, carboxymethylation, high-pressure homogenizer, rheology, cosmetic ingredients, natural cosmetic*

1. Introduction

Apples (*Malus sp., Rosaceae*) are one of the world's most popular and most consumed fruits.[1] It is known for its beneficial effects in preventing cardiovascular and respiratory diseases, diabetes, obesity, and cancer.[2,3] Most harvested apples are processed into juice and cider, and industrial

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1 Department of Forest Product, Graduate School, Chungbuk National University, Graduate Student
2 Department of Forest Product, Graduate School, Chungbuk National University, Undergraduate Student
3 Department of Biochemistry, Chungbuk National University, Professor
4 Department of Forest Product, Graduate School, Chungbuk National University, Professor
† Corresponding Author: E-mail: soojshin@chungbuk.ac.kr (Address: Department of Forest Product, Graduate School, Chungbuk National University, Cheongju Chungbuk, 28644, Republic of Korea)
‡ Co-corresponding Author: E-mail: haseung@chungbuk.ac.kr (Address: Department of Biochemistry, Chungbuk National University, Cheongju Chungbuk, 28644, Republic of Korea)

activities produce large quantities of unused byproducts.[4] The apple pomace (approximately 1/4 of the weight of the fresh fruit) that must be produced after juicing consists of the pulp, peel, pit, and stem of various apple varieties.[5] Although these residues are considered waste, they are rich in bioactive compounds, mainly polyphenols, fiber, vitamins, and carotenoids.[7,8] Although some polyphenols migrate into the juice during processing steps, most remain in the apple peel and the pomace.[6,8] Apple pomace component was 44.5–57.4% of total carbohydrate, 18.1–18.3% of glucose, 2.7–5.3% of protein, 3.2–13.3% of pectin, and 1.1–3.6% of fat.[9] Another apple pomace composition reported by Ma et al., were 17.7% cellulose, 19.6% pectin, 15.4% lignin, 10.9% hemicellulose, 1.9% ash, and 24.5% extraction.[10]

The antioxidant content of apple peel is 2 to 9 times that of pulp.[11–13] Ursolic acid, present in the wax layer on the surface of apples, is a triterpenoid, a physiologically active substance, not only anti-inflammatory, antibacterial, and antiangiogenic but also has cytotoxic and cardiovascular effects.[14,15] The effect of extracting apple on total polyphenol and flavonoid components affecting antioxidant, antibacterial, anti-inflammatory activity, and cytotoxicity was investigated, and suitability as a cosmetic material was reported.[16–18]

In addition to its anti-aging effects, identified by Park et al., an in vitro study also demonstrated the inhibitory capacity of a polyphenolic apple extract on the fat type produced in sebaceous cells,[19] suggesting that these secondary metabolites might regulate sebum production, relieve skin diseases such as acne,[20] and reduce dermal inflammation, a capacity attributed to their inherent antioxidant assets.[6,21]

The galacturonic acid content in apples extracted by the acid-base method is 69.0–73.8%, by the alkali method 76.1%, by neutral extraction method, and 76.8–77.5% by the acid method.[22] Analysis of the carbohydrate composition of the fraction obtained after solubilization, which consists of galacturonic acid (40–64 mol%), arabinose (14–23 mol%), galactose (6–15%), and a small amount of rhamnose, xylose, and glucose.[23,24] Apple fiber is composed of cellulose, hemicellulose, lignin, and gums. This bulking fiber has been reported to increase intestinal fluidity and fecal water content.[22]

Pectin is a polysaccharide found in the cell walls of fruits and vegetables.[25] It is an excellent stabilizer and thickener and can be used as a food or cosmetic raw material.[26] To extract pectin, it hydrolyzes insoluble pectin with a high-temperature acid solution and extracts it together with other water-soluble components such as pectin, neutral polysaccharides, gums, and tannins.[27]

Nanocellulose can be said to be cellulose particles/fibrils which have a width or diameter smaller than 100 nm.[28] apple pomace nanoparticles, as the fibrillated nanoparticles are often called, are primarily manufactured using different forms of mechanical shearing of wood fibres.[29] Here the rheological properties of the nanocellulose suspensions are of prime concern, and several studies have been on this subject.[28] In general, suspensions based on apple pomace nanoparticles exhibit complex rheological behavior already at low concentrations of the cellulose material.[28,30] They are shear-thinning, viscoelastic, and exhibit an extensional viscosity.[31]

Thus, this study intended to explore and describe the conversion of apple pomace to nanoparticles and its potential as an innovative source of cosmeceuticals to be included in new natural formulations, adding value to this material currently considered industrial bio-waste. In addition to this main purpose, an additional humectant characterization of its apple pomace hydrogel was also performed to assess potential cosmetic applications. Moreover, instead of using typical commercial gelling agents (such as gum and Carbopol 940), we intended to make hydrogel from apple pomace by carboxyl pretreatment and high-pressure homogenizer to confirm the possibility of being a new rheology enhancer.

2. Materials and Methods

2.1 Materials

This experiment was conducted using cultivars of apple, Arisu, Busa, and Gamhong, from Chungju Agricultural Technology Center, Korea. Chemicals used for pretreatment of the degree of substitutions (DS) were: ethanol (Samjeon Chemical, 99.0% purity), caustic soda (OCI Chemical, purity 99.0%), and monochloroacetic acid, (MCA, Denak Co, Ltd, Japan).

2.2 Preparation of apple pomace powder

The apples have been mashed with the skin and strained the juice. Collected the pomace, added ethanol until all sank, and left overnight. Filtered and dried precipitate in an oven at 105° C for 6 hours.

2.3 Preparation of apple pomace nanoparticles in different degrees of substitution

A carboxymethylation substitution reaction was carried out using 30.0 g of apple powder. Table 1 summarizes the amount of chemicals used to react with two different degrees of substitution. After carboxymethylation, the mixture was washed and filtered repeatedly using distilled water until a neutral pH was reached. The cellulose fibers were dried in an oven and ground to a fine powder.

2.4 Apple pomace nanoparticles cultivars DS gel preparation

After carboxymethylation, the treated apple powder was added to 2.0% distilled water to make a suspension, and then a homogenizer (IKA T25 Digital Ultra Turrax, Germany) with dispersing tool IKA S25KD-LR-25F was used at 6,500 rpm for 10 minutes. Then a high-pressure homogenizer (Panda PLYS 2000, GEA, Italy) was used to form nanofibers. The treatment conditions were passed through the device four times while maintaining a pressure of 600 bar to obtain a gel of 2% concentration of each hydrogel of apple pomace nanoparticles (APN) DS for the next experiment.

2.5 Water retention value (WRV)

The WRV of the samples was determined through centrifugation at 30℃ and 3000 G for 30 minutes using a type: H-103N centrifuge, a product of Kokusan Enshinki Co., Ltd Tokyo, Japan. The Apple Pomace Nanoparticles weighed 5 g and was put in a P4 (10-16 μm) WRV filter glass covered with a tea bag filter inside. After centrifugation, the leftover sample was removed and weighed to determine the weight of the centrifuge DS Apple. The samples were dried in an oven at 80℃ and left overnight. The WRV was calculated using the equation [1] below:

$$\mathrm{WRV}\ (\%) = \frac{\text{wet sample (g)} - \text{dry sample (g)}}{\text{dry sample (g)}} \times 100 \qquad [1]$$

2.6 Rheological characterization

The mechanical properties of apple pomace nanoparticle gel were characterized by a rheometer

Table 1. Pretreatment condition for carboxymethylation

Sample	Ethanol (ml)	NaOH (g)	MCA (g)	Apple (g)
DS 0.0	-	-	-	30
DS 0.2	200	4.44	4.33	30
DS 0.4	200	8.89	8.67	30

(MCR 102, Anton Paar, Austria). At 25℃, a parallel plate with a diameter of 25 mm and a 1 mm gap between the two plates were set to measure the shear viscosity and amplitude sweep. The viscosity of the sample was measured at a shear rate of 1 s^{-1} to 21 s^{-1}. The amplitude sweep was measured to determine the linear viscoelasticity (LVE) area at a 10 radians/sec frequency in the strain range of 0.01 to 25%. The flow point (strain γf) was evaluated when the storage modulus was equal to the loss modulus ($G'=G''$).

3. Result and Discussion

3.1 Apple pomace nanoparticles hydrogel as a humectant

Moisturizers play an important role in cosmetics. It helps to improve the skin condition by keeping the skin surface moist and not dry.[32] According to the moisturizing mechanism, moisturizers are divided into emollients in the fat with hydrophobic interaction and humectants that inhibit moisture evaporation through strong hydrogen bonding.[33] Substances containing a carboxyl group are capable of strong hydrogen bonding and can be used as a humectant-based moisturizer.[32, 33]

Water Retention Value (WRV) is an empirical measure test of the water holding capacity of a fiber sample.[34] As internal fibrillation, internal pore enlargement, and delamination (called “swelling”) occur simultaneously with the development of external fibrils, WRV increases with increasing beating (making smaller size),[35] which also helps retain additional water.[36] Add carboxymethylation pretreatment can improve nano fibrillation, increase the number of surface hydroxyl groups and add carboxyl groups that can increase WRV.[37] The higher WRV, the more fibers can hold water which can increase the ability of moisture.

Since saccharides have hydroxyl groups (pentose or hexose), if the polysaccharide structure can form a sufficient hydrogen bond with water as a solvent, it can be an excellent moisturizer candidate.[38] Water retention value is required to determine the moisturizing properties of apples, measured by the amount of water remaining after centrifugation under strong centrifugation conditions.[39] Products with excellent moisture retention are suitable as cosmetic moisturizers.

Water retention of nanofibers of apple powder treated with untreated (DS 0), substitution degree 0.2, and substitution degree 0.4 of three apple cultivars were also measured. As shown in Fig. 1, zeta potential can determine each apple cultivar’s carboxylate from pectin and carboxyl group content.[40] (Zahra, et al., 2022)

The DS 0 shows carboxylate from pectin in each apple cultivar and increases the degree of substitution, increasing the anion from the carboxyl groups attached to the APN hydrogel DS.[40] The increase of 14-23% of anion from carboxyl groups in carboxymethyl cellulose improved water retention as the degree of substitution increased compared to that of no treatment, as shown in Fig. 2. The increase in WRV in the DS apple pomace was twice that of the original apple pomace, indicating that the addition of the carboxyl group had a considerable effect, and the carboxylate of pectin had little effect.

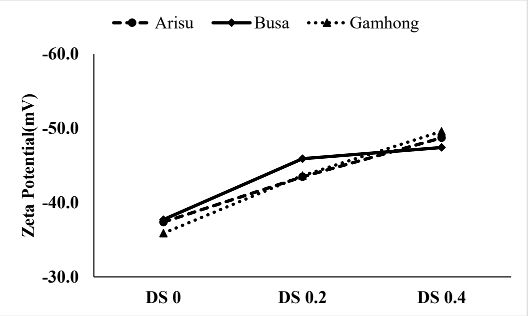

Fig. 1. Zeta potential of different DS of apple pomace nanoparticles for different cultivars.[40]

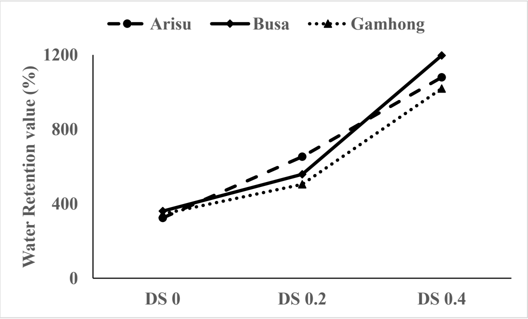

Fig. 2. Different water retention values from different DS-treated apple pomace nanoparticles for different cultivars.

At the substitution degree of 0.4, the WRV was highest because of the small nanofiber widths. This was thought to be due to the relatively increased hydrogen bonding with the water solvent because the small-width fibers had a large specific surface area. Based on the moisture retention rate in Fig. 2, the ones suitable for cosmetic moisturizers are nanofibers after pretreatment with a substitution degree of 0.4. Busa DS 0.4 has the highest WRV because has more uronic acid than other cultivars.[40] Meanwhile, the WRV of viscose nonwoven was around 65%, fiber alginate about 85%, and carboxymethyl cellulose (Na-CMC) was 1,500%,[41] it can be concluded that Busa APN hydrogel DS 0.4 was better than viscose nonwoven and alginate but less than Na-CMC (viscose<alginate<Busa DS 0.4 <Na-CMC).

Humectant moisturizers used by cosmetic companies include polyhydric alcohols such as glycerol, propanediol, butanediol, and hyaluronate (which contain a carboxyl group).[42] Sugars such as trehalose are also used as humectants.[43] The carboxyl group has stronger hydrogen bonding than hydroxyl group, and the exchange of the hydroxyl group with carboxyl group can increase the WRV, which acts as a humectant (shown in Fig. 2). We reported that the polysaccharides that makeup apples are composed of various monosaccharides and uronic acids, making them suitable as a moisturizer.[40]

3.2 Apple pomace nanoparticles hydrogel as a physical stability/consistency

The effects of carboxyl content were investigated using various apple pomace different cultivars and DS. The change in viscosity caused by the influence of the carboxyl attached to cellulose in the apple pomace is summarized in Figs. 3a-c. All of them show that viscosity increased than untreated. In Fig. 3, the carboxyl content attached to cellulose significantly affects the gel strength by increasing the viscosity. The viscosity of the hydrogel was increased because of the network structure formed

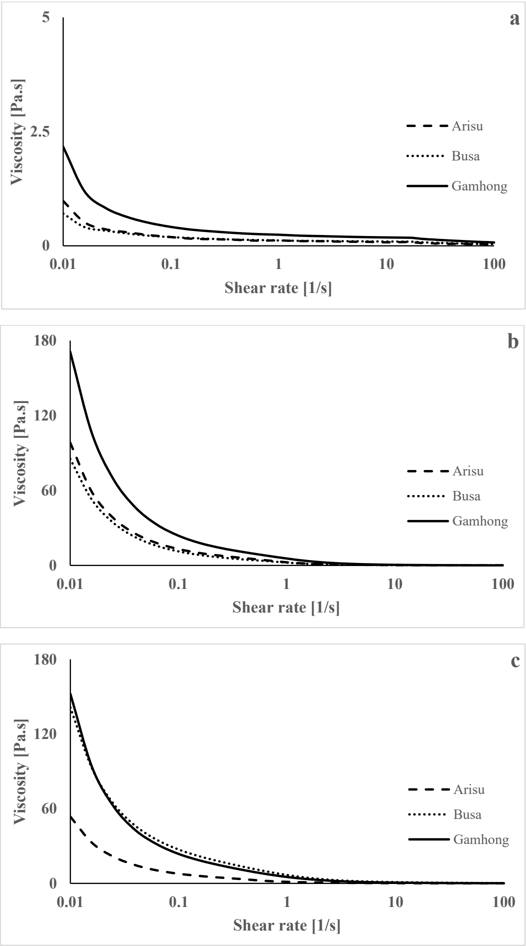

Fig. 3. Viscosity as a function of shear rate for 2% CNF Apple DS at various cultivars a) DS 0, b) DS 0.2, c) DS 0.4.

by crosslinking the water solvent and the negatively charged nanoparticle apple pomace (added carboxyl) with optimum carboxyl group content. All sample viscosity measured by at a shear rate of 1 s^{-1} to 100 s^{-1}.

Fig. 3c showed that all nanoparticle apple cultivars with DS 0.4 viscosity decreased than DS 0.2. However, APN hydrogel from DS 0.2 to DS 0.4 decreased the viscosity. These results show that the viscosity of nanoparticle apple pomace containing a carboxyl amount improves progressively when the concentration is increased from DS 0 to DS 0.2 and decreases when adding carboxyl up to DS 0.4; more carboxyl content made nanofibril smaller size,[40] that made the viscosity weaker. A similar trend result of carboxymethyl cellulose from papaya peel (CMCp) viscosity from 0.05 wt% (2,500 mPa·s) to 0.20 wt% (6,800 mPa·s) and decrease at 0.25 wt% (3,500 mPa·s) CMC.[44]

All the samples showed typical shear thinning behavior with a decrease in viscosity when the shear rate increases. An increase in the shear rate leads to a gradual breakdown in the network, which results in a higher viscosity in APN hydrogel from DS 0 to DS 0.2 than DS 0.4. The carboxyl group in cellulose contributed positively to viscosity, but pectin contributed not that well to viscosity.

In this study, nanoparticles of apple pomace from DS 0.2 substituted Gamhong cultivar had the highest viscosity (171,075 mPa·s) than Arisu and Busa. This might be attributed to the difference in cellulose and pectin content of each cultivar, where Gamhong has more cellulose and low pectin than Arisu and Busa, which had lower cellulose and higher pectin.[40] Meanwhile, 40% glycerol solution had 3.686 mPa·s,[46] 1.8% collagen had 12,800 mPa·s,[47] and 1% Carbopol 941 had 11,000 mPa·s,[45] 0.5% Carbopol 940 had 44,000 mPa·s (Kumara, et al, 2015), 0.5% Carbopol 980 had 65,000 mPa·s[48] and 1.6% Aristoflex AVC polymer had almost 70,000 mPa·s.[44] This shows that APN hydrogel DS 0.2 has a higher viscosity than glycerol, collagen, and thickening agents (Carbopol 940, 941, 980, and Aristoflex AVC). APN hydrogel were good as other commercial products.

APN hydrogel containing more cellulose has a higher viscosity than a lower amount at concentrations of DS 0.2. At higher concentrations (DS 0.4), Gamhong still had the highest viscosity, Busa the second-highest, and Arisu the lowest. These trends indicate that the impact of cellulose and pectin on the viscosity was directly correlated with the kind of shear rate and concentration of carboxyl.

3.3 Apple pomace nanoparticles hydrogel as a spreading ability

Hydrogel properties were determined by storage modulus (G′) and loss modulus (G″). Adding carboxyl groups into apple pomace and making them into nanoparticles of hydrogel increased the storage and loss modulus of the APN hydrogel (Figs. 4-6). The effect of carboxyl groups on the storage and loss modulus of 2% APN hydrogel cultivar Arisu DS 0.2 and 0.4 is shown in Fig. 4. Carboxyl groups that attached in cellulose increased from DS 0.2 to DS 0.4, and the G' value decreased from 277 to 117 Pa, confirming that it created a stronger APN hydrogel cultivar Arisu at DS 0.2.

This indicates that by increasing the concentra-

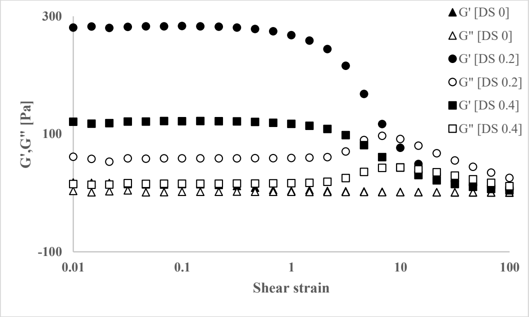

Fig. 4. Storage modulus and loss modulus as a function of strain amplitude at a frequency of 10 rad/s for 2% CNF Arisu Apple DS 0.2 and DS 0.4.

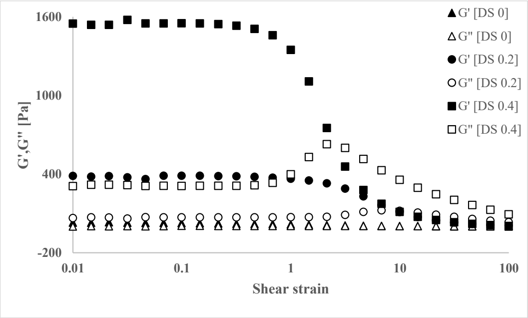

Fig. 5. Storage modulus and loss modulus as a function of strain amplitude at a frequency of 10 rad/s for 2% CNF Busa Apple DS 0.2 and DS 0.4.

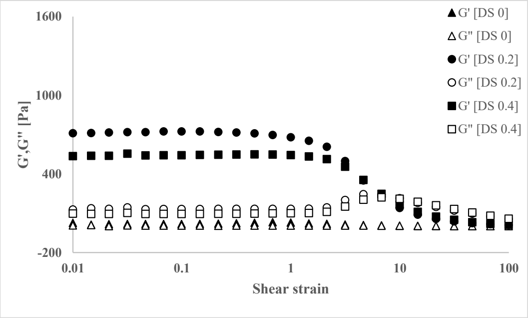

Fig. 6. Storage modulus and loss modulus as a function of strain amplitude at a frequency of 10 rad/s for 2% CNF Gamhong Apple DS 0.2 and DS 0.4.

tion of carboxyl groups, the gel hardens and becomes stronger until it reaches the upper limit of the APN hydrogel; further increases in the DS will reduce the strength of the hydrogel. The intimate contact between nanoparticles causes the stiffening of the gel due to the increased carboxyl content in the APN hydrogel. A similar trend was observed when the cultivar Gamhong was used, and the gel strength decreased when the concentration of carboxyl was increased from DS 0.2 to DS 0.4 (Fig. 6).

Fig. 5 shows the storage and loss modulus of the APN hydrogel cultivar Busa gel as a function of strain amplitude for various concentrations of carboxyl. The gel strength increases from DS 0.2 to DS 0.4 and reaches a maximum. Busa DS 0.4 formed the most potent gel (1,402.9 Pa). As shown, the cultivar Busa has a more significant effect on the hydrogel strength than Arisu and Gamhong (Busa > Gamhong > Arisu) at high concentrations (DS 0.4). Meanwhile, 1% Aristoflex AVC has around 300 Pa,[45] 0.5% Carbopol 940 has almost 400 Pa, and 0.5% Carbopol 980 has 500 Pa (Kumara et al., 2015). this shows that APN hydrogel Busa DS 0.4 and APN hydrogel Gamhong DS 0.2 (709 Pa) had a higher storage modulus than thickening agents (Carbopol 940, 980, and Aristoflex AVC).

4. Conclusions

We investigated the apple pomace cultivars prepared by carboxymethylation at different DS, followed by nano-processing. More carboxyl groups from pretreatment led to higher carboxyl content on the APN hydrogel contributed to enhancing WRV. Compared with commercial humectants or alginate, APN hydrogel had better WRV. Increased DS also significantly increased the viscosity at optimum at DS 0.2 and decreased at DS 0.4. APN hydrogel had better viscosity than glycerol or collagen. Storage and loss modulus had similar trends with viscosity of the hydrogels. APN hydrogel had storage and loss modulus as good as commercial thickening agents (Carbopol 940, 980, and Aristoflex AVC).

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