

# ANTIBACTERIAL ACTIVITY AND STORAGE STABILITY OF HYGIENIC WET WIPES BASED ON SUSTAINABLE NATURAL INGREDIENTS

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Wet wipes are good examples of common textile materials used in daily life. Although they can serve for different purposes, wet wipes for personal hygiene are especially popular, in particular, those with antimicrobial properties. Considering their demand, especially during epidemics, it is expected that the wet wipe market has the potential to expand further. In order to achieve antibacterial properties, wet wipes contain various chemicals, some of which can have a negative impact on human health. By using natural substances, it is aimed to minimize the use of harmful substances in wet wipes and offer innovative products on the market. For this, in the present study, leaf extract and fruit juice from gilaburu plants (*Viburnum opulus* L.) were used. The solutions obtained were used directly, without the addition of any further chemicals, in the impregnation of nonwoven webs/fabrics. Then, the impregnated nonwoven webs (wet wipes) were analyzed in terms of antibacterial properties and storage stability. The obtained results showed that the wipes (nonwoven webs) impregnated with gilaburu fruit juice could decrease *Escherichia coli* and *Staphylococcus aureus* bacteria populations even after 5 weeks of storage.

**Keywords:** personal hygiene, wet wipes, sustainability, natural ingredients, gilaburu (*Viburnum opulus* L.), antibacterial

## INTRODUCTION

The popularity of personal hygiene goods, including wet wipes, has been growing steadily, especially during epidemics, as one of the most important measures to be taken in such situations is to give major importance to cleaning and hygiene rules. In general, the global consumption of wet wipes is the result of modernization, and a large market has started to develop with the increase in consumer awareness after the 1990s.<sup>1-3</sup> Recent values exhibit a constantly increasing trend on this market. It was reported that the global wipes market had a value of \$39.6 billion in 2020, being expected to reach \$41.69 billion in 2021.<sup>4</sup>

Wet wipes can be defined as a general term including hand/body wipes, flushable wipes, baby wipes, facial/cosmetic wipes, etc. They may be composed of natural or synthetic fibers, but nonwoven wet wipes dominate the market at the moment.<sup>1-3</sup> Their increasing demand has stirred much research interest, as attempts have been

reported to achieve specific properties, especially antibacterial ones, by different approaches. For example, Devaki *et al.* developed bamboo-based wipes with the help of tulsi (*Ocimum tenuiflorum*) and clove (*Syzygium aromaticum*) extracts to control skin rashes and allergies caused by bacteria.<sup>5</sup> Fijak *et al.* tested mixtures comprising lactic acid, cocamidopropyl betaine, cetylpyridinium chloride, and benzalkonium chloride in different amounts for the wet wipe production to provide antibacterial protection.<sup>6</sup> In another study, four different methods were used in the preparation of silver nanoparticles that would be tested as a disinfectant in cellulose-based wipes. In terms of disinfectant performance and cytotoxicity, the techniques used in the preparation of silver nanoparticles were reported as determinant. Meanwhile, the benefits of using silver nanoparticles in terms of disinfection are widely known.<sup>7</sup> In a different study, *Melaleuca alternifolia* pickering emulsion stabilized by

cellulose nanofibrils has been reported as useful in the production of antimicrobial cotton wipes.<sup>8</sup>

One of the challenges in producing wet wipes is to ensure that they are protected against harmful effects until their use. Notably, wet and moist surfaces represent a favorable environment for yeasts, molds and bacteria to thrive. Because of this, the addition of preservative chemicals is an important way to protect the product and the consumer.<sup>9</sup> Moreover, additional chemicals can be preferred in the production of wet wipes to ensure different features. However, the use of certain chemicals in wet wipes has been reported to cause serious health issues. For example, Chang and Nakari stated that methylisothiazolinone in baby wipes leads to allergic contact dermatitis.<sup>10</sup> Aschenbeck and Warshaw identified 132 different substances from 54 different wet wipes, and stated the presence of potential allergens in hygienic wet wipes.<sup>11</sup>

This study attempted to produce sustainable hygienic wet wipes using cellulose-based nonwoven materials (webs). To gain antibacterial functionality, the webs were impregnated with herbal-based solutions of the gilaburu plant (*Viburnum opulus* L.) – fruit juice and leaf extract –, without using any preservatives or chemicals. The gilaburu plant (*Viburnum opulus* L.) is a member of the *Caprifoliaceae* family, is native to Europe and generally distributed in the Central Anatolian Region of Turkey. Its popular names in English are guelder rose and European cranberry bush.<sup>13</sup> In Turkey, gilaburu berries are traditionally used to prepare drinks, marmalades, jams, cakes, jellies and sauces.<sup>12</sup> The berries of *Viburnum opulus* L. have been found to have high levels of polyphenols, including tannins, anthocyanins, chlorogenic acid, (+)-catechin, (-)-epicatechin, cyanidin-3-glucoside, cyanidin-3-rutinoside, and quercetin.<sup>14</sup> Due to its rich content of polyphenols, it is also employed for medical purposes. Traditional uses for gilaburu juice include treating cough, colds, tuberculosis, rheumatoid arthritis, ulcers, liver illness, diabetes, and hypertension, as well as preventing some renal and stomach issues.<sup>15</sup> A significant number of research works have reported on the antibacterial,<sup>13,15-18</sup> antioxidant,<sup>15,19-23</sup> and anti-inflammatory<sup>15,24</sup> properties of gilaburu. Gilaburu fruit juice has been shown to be beneficial in the early stages of colon cancer and for prevention.<sup>25</sup> In another study, the probiotic properties of lactic

acid bacteria from fermented gilaburu fruit juice were reported as effective against certain pathogenic bacteria.<sup>26</sup> Additionally, *Viburnum opulus* L. was reported to help in reducing the side effects of taxane class chemotherapy drugs.<sup>27</sup> The stability of anthocyanins obtained from *Viburnum opulus* berries, which are responsible from some biological features, has been also studied.<sup>28</sup>

Due to these beneficial properties, especially the antibacterial ones, *Viburnum opulus* L. has also attracted some research attention in textile applications.<sup>17,18</sup> In this respect, it has been examined for imparting antimicrobial features to wool<sup>17</sup> and cotton<sup>18</sup> fabrics. Unlike previous studies, this work aimed to investigate the usability of gilaburu extracts in antimicrobial wet wipe production. For this purpose, the juice of gilaburu berries and the extract of dried and ground gilaburu leaves were tested to determine their efficiency in the development of cellulose-based wet wipes with antimicrobial properties.

## EXPERIMENTAL

### Materials

In this study, a cellulose-based (85% viscose and 15% cotton) non-woven material (web), with the weight of approximately 50 g/m<sup>2</sup>, was used for the production of wet wipes. The leaf extract and berry juice of *Viburnum opulus* L. were used as solutions for the impregnation of the wipes. Gilaburu fruits (packed in plastic jars containing water) and leaves (dried and ground) were obtained from local markets in Kayseri province. Figure 1 presents the gilaburu berry and ground leaves used in this study.

### Methods

Two different solutions were used in the experiments. First, the gilaburu plant leaf extract was obtained by the Soxhlet extraction method. In the Soxhlet extraction process, 20 grams of ground dry leaves were extracted with 500 mL of water for 4 hours to obtain the leaf extract. The other solution represented the juice of the gilaburu fruit, and it was obtained by directly squeezing the berries. These two extracts (solutions) were used separately to impregnate cellulose-based wipes to a water pick-up value (WPV) of 600%. The workflow for the production of experimental wet wipes is presented in Figure 2.

The physical properties of the experimental samples were determined before and after the impregnation step. Tensile strength measurements of the samples were performed on an Instron 4411 Tensile Strength Tester.

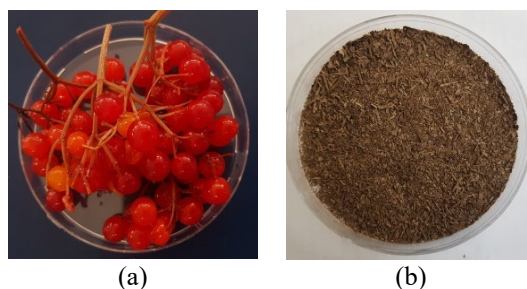


Figure 1: Berries (a) and ground leaves (b) of gilaburu (*Viburnum opulus* L.)

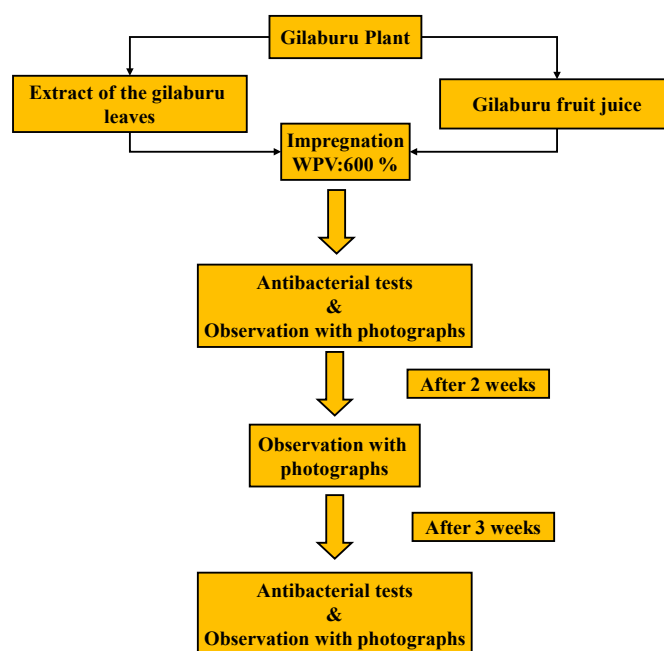


Figure 2: Workflow for experimental wet wipe production

## Characterization

### Antibacterial tests

The antibacterial properties of herbal extracts and webs impregnated with them were determined against *Escherichia coli* (ATCC 25922) and *Staphylococcus aureus* (ATCC 29213) bacteria, following the ASTM E 2149-01 standard.<sup>29</sup> The antibacterial activities were determined after 24 hours of contact time with the bacterial solution, as detailed in a previous study by Yılmaz.<sup>30</sup> Antibacterial tests were carried out directly after impregnation, and 5 weeks later, as shown in Figure 2.

### Analysis of storage stability and physical properties

For the analysis of storage stability, the samples were packed after impregnation and left for different durations in the dark at room temperature. The samples were photographed 2 weeks later and then again after 3 weeks – 5 weeks after impregnation (Fig. 2).

### FTIR analysis

Among the tested solutions, the Fourier transform infrared spectroscopy (FTIR) spectra were investigated only for the gilaburu fruit juice, as it exhibited a

prominent antimicrobial property. A Spotlight 400 FT-IR Imaging System (PerkinElmer) was used for this purpose. During the measurements, 32 scans with a resolution of  $4\text{ cm}^{-1}$  were acquired in the wavelength range of  $4000\text{--}400\text{ cm}^{-1}$ .

### Absorbance analysis

A UV-Vis spectrophotometer (PG Instrument T70) was used to measure the absorbance of the fruit juice, due to its prominent antimicrobial properties, to confirm the color of the samples. The absorbance was measured for  $380\text{--}720\text{ nm}$  with an interval of  $5\text{ nm}$ .

## RESULTS AND DISCUSSION

The samples immersed into the extract of gilaburu leaves and berry juice were analyzed in terms of three important parameters: their tensile strength, antibacterial activity and storage stability.

The tensile strength measurements of the samples revealed that the impregnation caused dramatic losses in tensile strength. The strength of the nonwoven samples in longitudinal direction

was determined as 64.6 N before impregnation and as 39 N immediately after impregnation. This dramatic strength loss is most probably related to the composition of the webs. It is well known that viscose fibers, which represent the major component of the nonwoven webs used in this study, have lower strengths in wet forms.

The initial nonwoven webs are presented in Figure 3 (a), along with a microscopic view (Olympus SZ61 stereo microscope) of the untreated samples (Fig. 3 (b)). After impregnation, the nonwoven wipes have changed color, which is most obvious when the gilaburu fruit juice is used for impregnation, as may be seen in Figure 3 (c). The wipes impregnated with

the fruit juice took a dominant reddish color, while those impregnated with the gilaburu leaf extract turned light brown (Fig. 3 (d)). It is obvious that the juice of the berries dramatically colored the nonwoven surfaces. Due to this significant coloration, the absorption spectroscopy curve of the juice was recorded and presented in Figure 4. The undiluted form of the juice in a cuvette is included in Figure 4, along with the absorbance–wavelength curve after dilution. The characteristic peak of the red color is observed in the spectrum, which confirms that the coloration of the wet wipes impregnated with the juice comes from the juice itself.

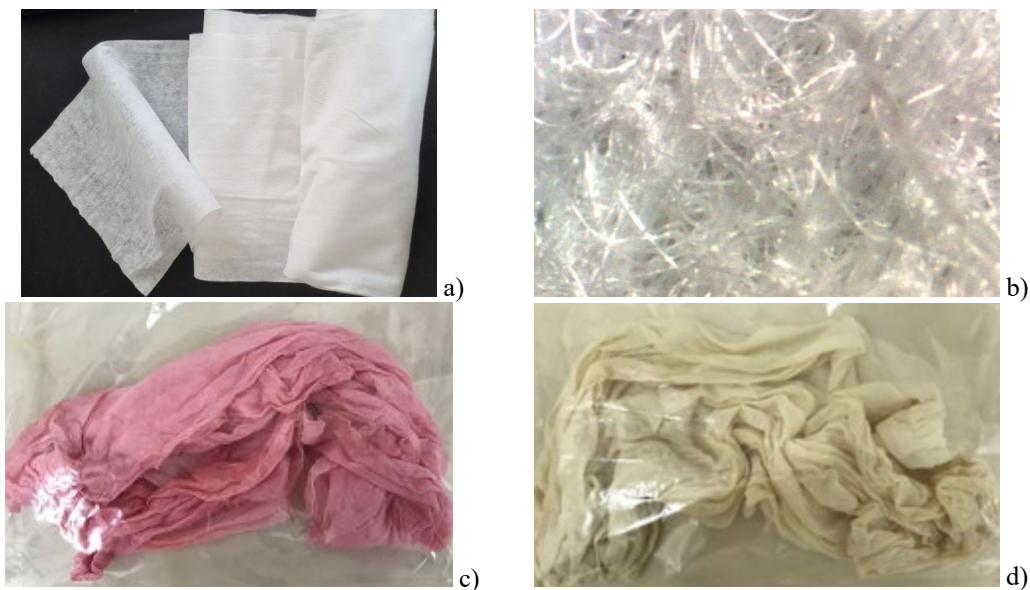


Figure 3: Nonwoven wipes before impregnation (a), along with their microscopic view at 40x magnification, and the samples impregnated with gilaburu fruit juice (c) and leaf extract (d)

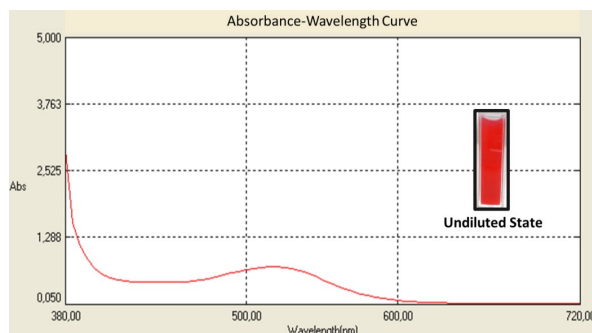


Figure 4: The absorption spectroscopy curve of the fruit juice after 1:5 dilution

### Analysis of wet wipes in terms of antibacterial activity

In the literature, a significant number of studies are available on the use of various herbal sources in finishing textiles for imparting antimicrobial and other functional properties. For

example, thyme oil was reported as a biocide for protection of linen and mixed linen/cotton materials,<sup>31</sup> an extract of olive tree leaves for antibacterial finishing of cotton,<sup>32</sup> lemongrass oil for fragrance, antibacterial and antioxidant activity in cotton treated with  $\beta$ -cyclodextrin-

grafted chitosan,<sup>33</sup> *Bombax ceiba* barks for mosquito repellency, antibacterial and UV protection of cotton.<sup>34</sup> Also, significant antibacterial activities have been observed for the juice and leaf extract of *Viburnum opulus* L.<sup>13,15-18</sup>

In the light of these findings, our study was motivated by the idea of preparing antibacterial wet wipes. The cellulose-based nonwoven webs were impregnated with gilaburu fruit juice and leaf extract, and then the obtained wet wipes were tested in terms of antibacterial activity against *Escherichia coli* (ATCC 25922) and

*Staphylococcus aureus* (ATCC 29213). For this purpose, the ASTM E2149 standard<sup>29</sup> was followed, which addresses the evaluation of antimicrobial features of textiles. The obtained antimicrobial activities of the wet wipes padded with gilaburu fruit juice and leaf extract are presented in Table 1. The data in Table 1 reveal an antibacterial activity of 99% against *Escherichia coli* and *Staphylococcus aureus*, tested immediately after impregnation of the wipes. These results highlight the potential of such gilaburu extracts in production of wet wipes.

Table 1  
Antibacterial activities of nonwoven wipes after impregnation with gilaburu fruit juice and leaf extract

Impregnated with gilaburu fruit juice		Impregnated with gilaburu leaf extract	
Inhibition of			
<i>E. coli</i>	<i>S. aureus</i>	<i>E. coli</i>	<i>S. aureus</i>
99%	99%	99%	99%

#### Analysis of wet wipes in terms of storage stability

Another important parameter that should be taken into account during the evaluation of wet wipes is their storage stability. This is especially important in this case, since only berry juice or leaf extract has been used for impregnating the wipes, without any chemical preservatives.

The observation during the storage period has been carried out in two directions: the first focused on visual observation of the appearance of the wipes, and the other – on the analysis of the antimicrobial feature, to determine how these features varied over time. For this purpose, the nonwoven wipes impregnated with the extract or juice at a pick-up value of 600% were placed into ziplock bags, to prevent loss of moisture, and left in the dark at room temperature. The samples were photographed periodically, as presented in Figure 5.

Figure 5 shows color changes occurring in the wipes during the storage period. In the case of using the juice of gilaburu berries for impregnation of the wipes, after two weeks of storage, the red color lightened and then, after an additional three weeks, it turned to a purple-brown color. Meanwhile, the color of the samples impregnated with the leaf extract was not affected so much by the storage period.

Beyond these color changes, mold growth has been considered as an important parameter. It was observed that the nonwoven samples impregnated with the leaf extract became moldy after 2 weeks

of storage, while no mold growth was observed on the samples impregnated with fruit juice. Later, 5 weeks after impregnation, the wet wipes were observed repeatedly, and again, no mold was found on the samples impregnated with fruit juice, while on the others, there was increased mold development, compared to their previous aspect (after 2 weeks of storage).

Additionally, the stability of the antibacterial activity of the samples during storage has been examined. For this purpose, the antibacterial activities against the same strains of *Escherichia coli* and *Staphylococcus aureus* were tested after 5 weeks of storage. As expected, in accordance with the moldy surface, the samples impregnated with the extract of the gilaburu leaves showed no antibacterial activity against the tested bacteria. However, the samples impregnated with gilaburu fruit juice provided antibacterial activity, even after 5 weeks of storage. The bacterial inhibition was found to reach 70% and 99% for *E. coli* and *S. aureus*, respectively. Otherwise said, a decrease in antibacterial activity was observed against *E. coli*, while there was still significant antibacterial activity against *S. aureus*. This antibacterial profile of the gilaburu fruit juice was reported to be related to the presence of some specific metal ions and phenolic compounds in its composition, which are responsible for the bactericidal effects.<sup>18</sup>

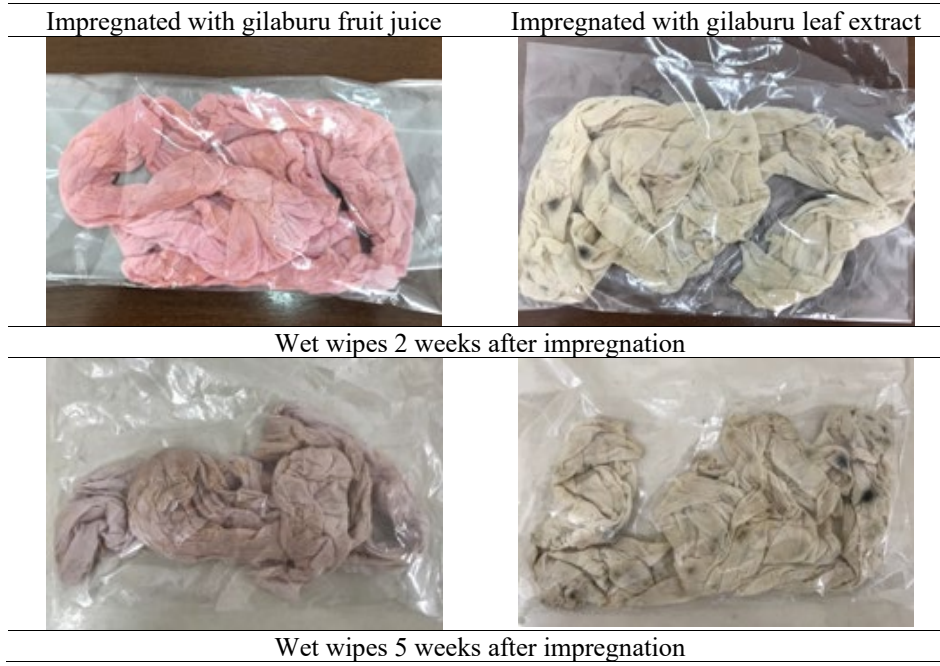


Figure 5: Variation of color of impregnated wet wipes during the storage period

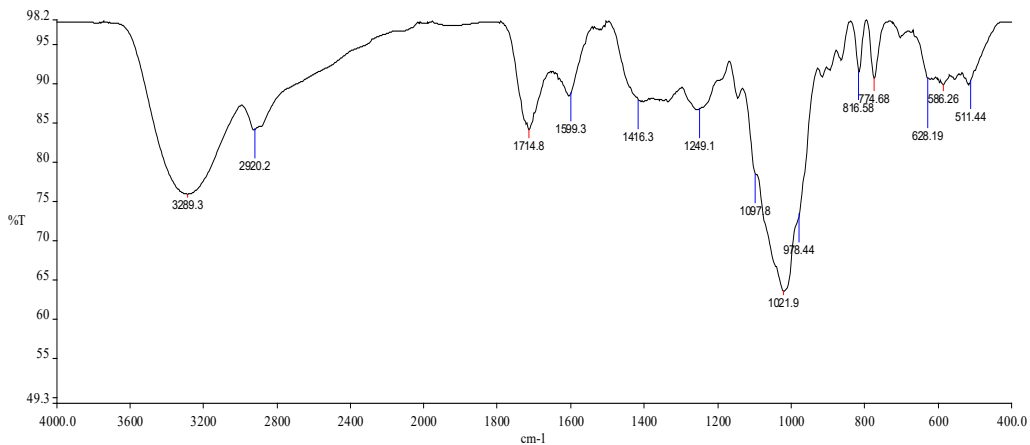


Figure 6: FTIR spectrum of gilaburu fruit juice

Considering this prominent antimicrobial property exhibited by the gilaburu berry juice padded wipes, FTIR analysis was performed to investigate the composition of the juice. For this, the juice sample was subjected to lyophilization at first. The obtained spectrum of the gilaburu berry juice is presented in Figure 6. The FTIR spectrum in Figure 6 shows the most prominent peaks at 774.7  $\text{cm}^{-1}$ , 816.6  $\text{cm}^{-1}$ , 1021.9  $\text{cm}^{-1}$ , 1249.4  $\text{cm}^{-1}$ , 1416.3  $\text{cm}^{-1}$ , 1599.3  $\text{cm}^{-1}$ , 1714.8  $\text{cm}^{-1}$ , 2920.2  $\text{cm}^{-1}$  and 3289.2  $\text{cm}^{-1}$ .

A number of studies have focused on the FTIR spectroscopy of *Viburnum opulus*. It is clear that some shifts in the bands can be caused by the extraction process of the juice. In the present study, the juice of the gilaburu was obtained by just squeezing the berries, but the spectrum (Fig.

6) agrees with previous results in general.<sup>35-41</sup> Thus, Ildiz *et al.* studied the FTIR spectrum of *Viburnum opulus* L. and reported the range of 500~538  $\text{cm}^{-1}$  as a broad and weak bending vibration for the O-P-O groups in the juice.<sup>36</sup> In our study, the peak at 511  $\text{cm}^{-1}$  can be given this assignment. Additionally, the peaks at 1717  $\text{cm}^{-1}$  and 3256  $\text{cm}^{-1}$  were reported as assigned to the C=O group and OH group of phenolic compounds.<sup>36</sup> Similar peaks were observed for the tested fruit juice in this study – at 1715  $\text{cm}^{-1}$  and 3289  $\text{cm}^{-1}$ , respectively. This confirms the presence of phenolic compounds in the juice, which can explain the outstanding antimicrobial activity of the fruit juice tested in this work. Additionally, the peak at 1249  $\text{cm}^{-1}$  was assumed to be related to the C-O stretching vibration of the



phenolic structure, which was observed at 1260  $\text{cm}^{-1}$  by Özdemir *et al.*<sup>40</sup> and at 1236  $\text{cm}^{-1}$  by Moldovan *et al.*<sup>37</sup> Moreover, the band at  $\sim 1599 \text{ cm}^{-1}$  in Figure 6 can be attributed to the C=C stretching vibration and the stretching bands of C-H were located at  $\sim 2920 \text{ cm}^{-1}$  for the tested juice in the light of findings by Moldovan *et al.*<sup>37</sup> The peaks at 1456  $\text{cm}^{-1}$  and 1029  $\text{cm}^{-1}$  were attributed to oligosaccharides and glucose, respectively.<sup>41</sup> Thus, the peak at 1416  $\text{cm}^{-1}$  in the spectrum in Figure 6 can be attributed to the C-O bond of oligosaccharides, and the band at 1022  $\text{cm}^{-1}$  can be related to the glucose of the fruit juice.

## CONCLUSION

Today, wet wipes have become an essential item in our daily life. While a multitude of ingredients can be used to produce a wide variety of products for this rapidly developing global hygienic wet wipes market, many chemicals in their composition have been reported to cause various health problems depending on the area of usage. Therefore, finding gentle solutions both towards human health and towards the environment is essential in the production of wet wipes.

For this reason, this study examined the efficiency of *Viburnum opulus* fruit juice and leaf extract impregnated into cellulose-based nonwoven webs in imparting antimicrobial properties to them. The storage stability of the experimental wet wipes was also examined. Of the two solutions tested for impregnation, the fruit juice was the only one that exhibited both antibacterial activity and storage stability over 5 weeks. It is expected that these findings may provide a starting point in the development of wet wipes based on natural extracts, reducing the need for harsh chemicals, which may be harmful to the skin. Further studies are necessary in order to obtain longer storage stability and ensure dermatologic compatibility of the wet wipes. Also, combinations with dermatologic oils may be investigated to develop specific skincare products.

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