PRELIMINARY STUDY ON FORMULATION OF PVA-BASED FACE MASK INCORPORATED WITH SUGARCANE BAGASSE MICROFIBRILLATED CELLULOSE

DZUN NORAINI JIMAT[1*], ISTISYHAD MOHAMAD[1], AZURA AMID[2], AZLIN SUHAIDA AZMI[1], MOHD FIRDAUS ABD. WAHAB[1]

[1]Department of Biotechnology Engineering, Kulliyyah of Engineering, International Islamic University Malaysia (IIUM), Jalan Gombak, Malaysia
[2]International Institute for Halal Research and Training, International Islamic University Malaysia (IIUM), Jalan Gombak, Malaysia

**jnoraini@iium.edu.my*

***ABSTRACT*:** Cellulose facial masks have gained a huge interest in the cosmetic industry. Cellulose can be extracted from plant biomass, bacteria and algae. In this study, several formulated PVA-based facial masks (F1, F2, F3, F4) incorporated with microfibrillated cellulose extracted from sugarcane bagasse (MFC-SCB) were prepared. The concentration of polyvinyl alcohol (PVA) was varied (5%–20% (w/w)) while the concentration of microfibrillated cellulose of sugarcane bagasse (MFC-SCB) was fixed at 5% (w/w) to get the appropriate composition of the facial masks. The MFC-SCB was extracted through chemical treatment assisted with ultrasonication. Sensory tests in terms of adhesion to the skin, spreadability, color, odor, and drying time were performed. These tests were carried out by requesting the volunteers to rate the performance of the masks. The results showed that the formulated facial mask F3 (15% [w/w] of PVA and 5% [w/w] of MFC-SCB) has the highest average score (13.9) which is 82% from the total score compared to other formulated masks. However, the standard formulation mask F5 (15% [w/w] of PVA and 5% [w/w] of sodium carboxylmethyl cellulose, CMC) achieved the highest score (13.5) compared to F3 (12.5). The findings of this study proved that the presence of MFC-SCB with PVA has a competitive performance with the standard facial mask formulation.

KEYWORDS: *PVA-based face mask, Sugarcane bagasse, Microfibrillated cellulose, Adhesion, and Spreadability.*

1. INTRODUCTION

Facial mask is one of the cosmetic products that mainly serves for quick and deep moisturizing skin restoration. This cosmetic product has endured a great makeover with new formulations and perspectives for skincare. Many consumers are looking for a cosmetic product which is natural, toxic-free, effective, and has an easy and fast effect on the skin. Natural polymers such as cellulose pulp, hydrogel (silk and collagen), and cellulose nanofibers (biocellulose) and synthetic polymers such as poly(vinyl alcohol) and silicone are among the basic ingredients used in the formulation of facial mask [1- 4]. Facial mask normally presents in the form of hydrogels because it can provide high water content and effectively delivers the active ingredients to the skin [5]. However, it is mechanically weak. A study reported that cellulose masks are easy in handling and applying them due to their

greater mechanical properties compared to hydrogel masks [4]. Cellulose is the most abundant and renewable natural polymer, which is the main component of the plant-based materials. Due to its biodegradability, biocompatibility, and non-toxic features, it becomes one of the appealing materials in many areas such as textile industry, high-quality paper production, food, pharmaceutical and medical devices, electronics, and cosmetic. Furthermore, it has high water capacity and forms strong gels at low concentrations due to a larger number of hydroxyl groups that are good in maintaining a moist environment. Consequently, it suits as a moisturizing agent in cosmetic products in maintaining the moisture level of skin as well as an appropriate carrier to deliver active ingredients to the skins.

The adhesion property of a mask is due to the mechanical property of nanocellulose associated with the composition of the formulation that includes hydrophilic and hydrophobic chemicals which provide a good interface between the skin and the mask [4]. Cellulose microfibril (MFC) is an aggregated form of cellulose where the outer layer of the cellulose fibers has been stripped away by mechanical shearing thus, exposing the fibril bundles [6]. These fibrils are much smaller compared to the original fibres and their aggregation could apply in various cosmetic products. For an instance, the uneven shape/morphology of these aggregates benefits in giving soft outcome to the skin [7]. This result might be due to the diffusion transmission and reflection of light minimizing the appearance of the skin imperfections [7]. Solid features of MFC aggregates are also essential for application in skin peeling scrubs for cleansing cosmetic products [7].

Therefore, in this study, due to the unique properties of nanocellulose large surface area, high specific strength, and distinctive optical properties, it was chosen as one of the facial mask ingredients. The MFC was extracted from sugarcane bagasse (SCB) via chemical treatment assisted with ultrasonication as described in a previous study [8]. Nanocellulose of sugarcane bagasse (MFC-SCB) was then included in polyvinyl alcohol (PVA) which is the main material used in the formulation of facial mask. The hydrophilic nature of PVA that consists of vinyl alcohol as a repeating unit can form the linkage yielding the hydrogels without the help of chemical crosslinking agents [9]. It results in the formation of a film on the skin surface. This study aimed to use MFC extracted from sugarcane bagasse (SCB) in preparing PVA-based facial masks. The concentrations of polyvinyl alcohol (PVA) were varied to get the appropriate composition of the facial masks. Sensory tests were conducted in terms of color, odor, spreadability, adhesion to the skin, and drying time of the facial mask gel. The formulated facial mask with the highest score was also compared with the standard facial mask (F5) which used sodium carboxylmethyl cellulose (CMC) and F6, facial mask with 7% (w/w) of MFC-SCB.

2. MATERIALS AND METHOD

2.1. Materials

The agricultural residues, sugarcane bagasse (SCB) was obtained from Sri Gombak market, Selangor. Sodium hydroxide (NaOH) pellets, 35% hydrogen peroxide (H_2O_2), 95% sulfuric acids (H_2SO_4), polyvinyl alcohol (PVA, M_w: 50 kDa), glycerine, and sodium carboxymethyl cellulose (CMC) were purchased from Sigma-Aldrich. Ethanol (HmbG chemical) was purchased from Merck (Malaysia). Kaolin clay (cosmetic grade) was online purchased from Lazada.

2.2. Methods

2.2.1. Formulation of facial mask

Nanocellulose used in this study was extracted from sugarcane bagasse as described by [8] and the formulation of face masks was according to the standard formulation of PVA based face mask by Rigano (2014) with minor modification [1]. Alumina-based fine ceramic was replaced with Kaolin clay powder and rose essential oil was used as a fragrance. No preservative was added in the formulation of facial mask. Different concentrations of PVA in the range of 5–20% (w/w) were used in the ingredients (Table 1). The amount of SCB nanocellulose was kept constant at 5% (w/w). For standard formulation, 15% (w/w) of PVA and 5% (w/w) of CMC were used. A total of 5 mL of each formulated facial mask was spread evenly on a Petri dish and its weight was recorded priorly. The petri dish was placed in a desiccator at the room temperature. The weight of all prepared samples was recorded every 5 minutes for a 30-minutes period. The data was presented in a graph of mean weight of the sample versus time.

Table 1: Formulation of facial masks incorporated with nanocellulose of sugarcane bagasse (MFC-SCB)

5% (w/w) MFC-SCB Formulation	PVA (% (w/w))
F1	5
F2	10
F3	15
F4	20

2.2.2. Sensory tests of the prepared PVA-based facial masks incorporated with MFC-SCB

The size of dried facial mask gel was prepared in 2 × 2 cm. Four volunteers (women, in the range of 21–23 years old) evaluated the mask properties. The mask gel was placed at the forehead region for 30 minutes, and the process was repeated twice. The volunteers were required to rate their satisfaction on a four-point scale (excellent = 4, good = 3, fair = 2, poor = 1) based on the color, odor, spreadability, adhesion to the skin, and drying time of the facial mask gel. The summary of the rating scale of the parameter evaluated by the volunteers is shown in Table 2.

Table 2: Scoring score used for sensory tests of all formulated PVA-based facial masks incorporated with MFC-SCB

Parameter	Scale			
	Excellent (4)	**Good (3)**	**Fair (2)**	**Poor (1)**
Color (A)		Colored	Colorless	
Odor (B)		Pleasant	Odorless	
Spreadability (C)	Easy application	Very easy	Watery	Very sticky
Adhesion to the skin (D)		Less	Mild	Strong
Drying time (min) (E)	<40 min	<30 min	<20 min	<10 min

2.2.3. Comparison analysis

Formulation of facial mask with the highest score (F3) was compared with other formulated masks: standard formulation, 15% (w/w) of PVA and 5% (w/w) of sodium carboxylmethyl cellulose (CMC), and 15% (w/w) of PVA and 7% (w/w) of SCB nanocellulose as shown in Table 3.

Table 3: Formulation of facial masks incorporated with nanocellulose of sugarcane bagasse (MFC-SCB) used for comparison analysis

PVA (15% (w/w)) Formulation	**Cellulose (% (w/w))**
F3	5
F5 (standard)	5
F6	7

3. RESULTS AND DISCUSSION

3.1. Drying time

The weights of all formulated facial masks were observed for 30 minutes and the changes are shown in Fig. 1. No significant weight difference was observed. F3 samples had the lowest weight. *In vitro* drying time of facial mask specifically the peel-off type is essential to ensure that it is easily removed from local application.

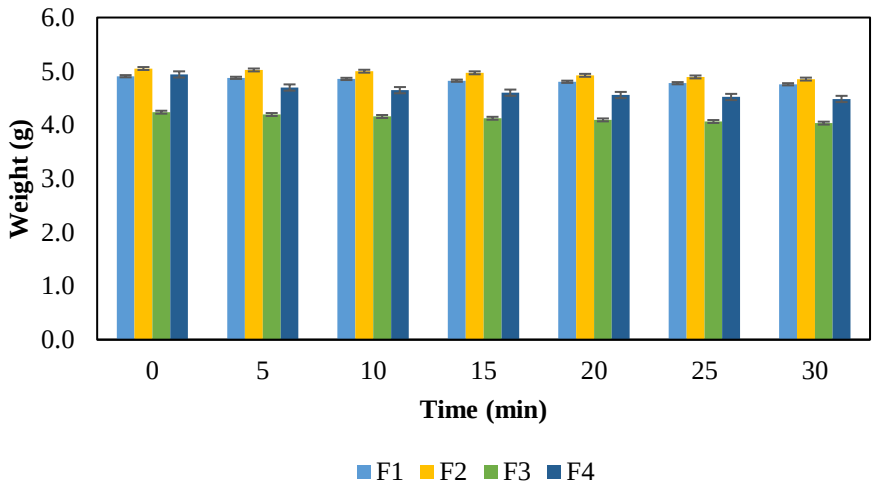

Fig. 1. The weight of facial mask for 30 minutes of drying time at the room temperature.

3.2 Evaluation of PVA-based facial mask incorporated with MFC-SCB

The evaluation was made based on the acknowledgement of the product performance and translated sensation into objective data which helped in studying the development of new products to assure the performance meets the requirements of the consumers. Table 4 shows the results of the sensory test for all PVA concentrations. For the color of the mask, all volunteers agreed that it is colorless for all formulated facial masks because no color additive was added and was left with the natural color of the mask. In terms of mask odor, almost all of the formulated facial masks gave a pleasant smell to the volunteers. Next, the spreadability of the samples might affect the adhesion to skin. If it is easy to spread, then it might stick to the skin nicely. As shown in Table 4, F1 sample was watery, probably due to

the low PVA concentration that lead to a less viscous solution, and consequently, it could not stick to the skin well. This situation is not favorable for a facial mask because it should adhere to the skin tightly but not to the extent irritating the skin [1]. Since the formulated masks were in the gel form, it could be peeled off after it dried but stronger adhesion to the skin surface would make it hard to peel. Therefore, it may irritate people with sensitive skin type. Overall, all formulated samples showed good result in terms of adhesion to the skin. This property is important as it reflects a good interface between the mask and the skin. It also indicates the mechanical properties of the mask such as its high malleability that relates to its blend formulation.

Table 4: Results of sensory tests of the all mask formulations by the volunteers

												RUN #1											
Subject	**F1**						**F2**						**F3**						**F4**				
	A	**B**	**C**	**D**	**E**	**Tot**	**A**	**B**	**C**	**D**	**E**	**Tot**	**A**	**B**	**C**	**D**	**E**	**Tot**	**A**	**B**	**C**	**D**	**E**	**Tot**
1	3	2	2	3	2	12	3	3	2	2	2	12	3	3	3	1	2	12	3	3	3	1	3	13
2	3	3	2	3	4	15	3	3	4	3	2	15	3	3	3	3	3	15	3	3	3	3	2	14
3	3	3	2	3	2	13	3	3	2	3	3	14	3	3	3	3	3	15	3	3	2	3	3	14
4	3	3	2	3	4	12	3	3	4	1	2	13	3	3	3	3	3	15	3	3	4	3	3	16
5	3	3	2	3	3	14	3	3	2	3	2	13	3	3	3	3	2	14	3	3	2	3	3	14
	Average score					13.2						13.4						14.2						14.2
												RUN #2											
1	3	2	2	1	3	11	3	3	4	2	3	15	3	3	3	1	2	12	3	3	2	1	2	11
2	3	3	2	3	4	15	3	3	4	3	2	13	3	3	3	3	3	15	3	3	3	3	3	15
3	3	3	2	3	2	13	3	3	2	3	2	13	3	3	3	3	1	13	3	2	2	3	4	14
4	3	3	2	3	4	15	3	3	4	1	3	14	3	3	3	3	2	14	3	3	4	3	4	17
5	3	3	2	3	3	14	3	3	2	3	2	13	3	3	3	3	2	14	3	3	2	3	3	14
	Average score					**13.6**						**13.6**						**13.6**						**11.6**
	Total average score					**13.4**						**13.5**						**13.9**						**12.9**

Drying time on the skin surface is not the only factor for selecting the best PVA concentration. Skin type plays an important role in determining the time taken for the formulation to dry. The drying time for people with moist/normal skin type may be longer compared to that for people with dry skin type. The rule of thumb of facial mask is to leave them on for a maximum of 20 min for the moisture and all the ingredients to sufficiently deliver to the skin [10]. The acceptability of the masks was evaluated through the subjective responses from the volunteers after applying the masks. Based on the rating score of the performance evaluation tests, F3 samples showed the highest average score (13.9) which is 82% from the total score compared to other formulated facial masks. However, to make the result more reliable, the tests should be performed with more volunteers.

3.3 Comparative analysis

All the three formulations F3, F5, and F6 were compared in another sensory test evaluation as shown in Table 5. The standard face mask formulation, F6 with 5% (w/w) of CMC was observed to be difficult to spread onto the skin surface and took a slightly longer time to dry compared to other two formulated face masks. This result might be due to the additional MFC-SCB that caused the mask to be too viscous and difficult to spread onto the skin and took a long time to dry. F3 formulation showed a mild adhesion to the skin compared to the other two formulations. The F5 sample achieved the highest score (13.5) compared to F3 samples (12.5), which is 7% higher. Meanwhile F6 has an average score of 10.5. However, this result shows that the F3 formulated mask could give an almost similar performance like CMC in the standard formulated mask.

Table 5: Sensory test for the facial masks with three different formulations

Formulation	Parameter					
	Color	Odor	Spreadability	Adhesion to skin	Time to dry on skin (min)	Total Score
			#Run1			
F3	2	3	3	2	2	12
F5	2	3	3	2	3	13
F6	2	3	1	1	3	10
			#Run2			
F3	2	3	4	2	2	13
F5	2	3	3	3	3	14
F6	2	3	1	1	4	11

4. CONCLUSION

Facial mask F3 (5% (w/w) of MFC-SCB and 15% (w/w) of PVA) is the best formulation achieved in this study based on its highest sensory test score. This formulation scored 7% less in the sensory test compared with the standard formulation mask F5 (5% (w/w) of CMC and 15% (w/w) of PVA). It can be concluded that MFC-SCB-based face mask showed a competitive performance in comparison to the standard mask formulation that uses CMC.

ACKNOWLEDGEMENT

This research is fully supported by FRGS grant (FRGS 16-044-0543). The authors fully acknowledged the Ministry of Higher Education (MOHE) and International Islamic University Malaysia (IIUM) for providing the equipment, chemicals, and instruments for this research.

REFERENCES

[1] Formulating facial masks Cosmet Toilet [https://www.cosmeticsandtoiletries.com/formulating/category/skincare/Formulating-Facial-Masks-275439021premium.html.]

[2] Suetake T, Sasai S, Zhen Y, Tagami H. (2000) Effects of silicone gel sheet on the stratum corneum hydration. British Journal of Plastic Surgery, 53:503–507. https://doi.org/10.1054/bjps.2000.3388

[3] Boonme P, Amnuaikit Chusuit, Raknam. (2011) Effects of a cellulose mask synthesized by a bacterium on facial skin characteristics and user satisfaction. Medical Devices: Evidence and Research, 77–81. https://doi.org/10.2147/mder.s20935

[4] Pacheco G, de Mello CV, Chiari-Andréo BG., Isaac VLB, Ribeiro SJL, Pecoraro É, Trovatti E. (2017) Bacterial cellulose skin masks—Properties and sensory tests. Journal of Cosmetic Dermatology, 1–8. https://doi.org/10.1111/jocd.12441

[5] Surini S, Auliyya A. (2017) Formulation of an anti-wrinkle hydrogel face mask containing ethanol extract of noni fruit (*Morinda citrifolia L*) for use as a nutracosmeceutical product. International Journal of Applied Pharmaceutics, 9: 74–76. https://doi.org/10.22159/ijap.2017.v9s1.41_47

[6] Pinkney S, Skuse D, Rowson N, Blackburn S. (2012) Microfibrillated cellulose- a new structural material.

[7] Why will MFC show potential in cosmetic applications in 2016 [https://www.exilva.com/blog/why-will-mfc-show-potential-in-cosmetic-applications-in-

2016]

[8] Asem M, Nawawi WMFW, Jimat DN (2018) Evaluation of water absorption of polyvinyl alcohol-starch biocomposite reinforced with sugarcane bagasse nanofibre: Optimization using Two-Level Factorial Design. IOP Conference Series: Materials Science and Engineering, 368(1). https://doi.org/10.1088/1757-899X/368/1/012005

[9] Lam NT, Chollakup, R Smitthipong, W Nimchua, T, Sukyai P. (2017) Utilizing cellulose from sugarcane baggase mixed with poly(vinyl alcohol) for tissue engineering scaffold fabrication. Industrial Crops & Products, 100: 183–197.

[10] How Long Should I Wear A Sheet Mask? 7 Important Things You Need To Know About This Popular Korean Beauty Product [https://www.bustle.com/articles/70368-how-long-should-i-wear-a-sheet-mask-7-important-things-you-need-to-know-about]