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Initial Investigation on Binding Agents for Compact Powder Formulation from Rice (*Bedak Sejuk*)

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ABSTRACT

Bedak sejuk is a type of traditional cosmetic product that is favoured by women that possesses potential to be marketed as cosmetic product in the form of loose powder or compact powder. At present, there is no research available on the bedak sejuk in the compact format. Therefore, the main objective of this study is to produce a compact powder by introducing several binding agents. The soaking process of rice grain in the water was carried out for 84 days (14 days soaking interval, 6 times soaking water changed) to produce the paste of bedak sejuk. It was then dried and crushed using mortar and pestle before being sieved to be characterizes (particles sizes and bonds- FTIR). The compact format of bedak sejuk were prepared by addition of different binding agents namely ethanol, isopropyl alcohol, polyvinyl alcohol(PVA), magnesium stearate and jojoba oil to evaluate its cracking level, hardness through drop test and easiness of application onto the skin. Based on the result, the particle size of uncrushed and crushed bedak sejuk were at 1000-2000μm and ranging 150-250 μm respectively. The FTIR analysis showed that the functional groups existed in bedak sejuk were OH bond, C-H bond, C=O bond, C-O-H bond and C-O bond. Each type of compact bedak sejuk produced from each binding agent exhibits their own strengths and weaknesses respectively. In short, alcohol (PVA) is found to be the best binding agent used due to its easiness to be applied onto the skin.

Keywords: Bedak sejuk; fermentation; binding agent; drop test; compact powder

INTRODUCTION

Bedak sejuk, which is also known as cooling powder, is a type of traditional cosmetic product using rice grain as the main ingredient and is still being used by the women in Malaysia. It is used as facial mask by mixing it with little amount of water, and then applied on the face, usually before going to sleep (Dzulfakar 2016). It gives a cooling effect to the user and is believed that *bedak sejuk* help to smoothen, whiten and tighten the skin (Sulaiman, 2015). It also uses to cure pimples and rashes, reduce the formation of oil on skin, remove dead skin cells and relieve itchiness caused by chickenpox (de Paepe 2002). Rice is reported to be rich in vitamins (B, E) (Satoshi et al. 2010), amino acids (Johar et al. 2018; Hasniza & Noorhisham, 2019) and antioxidants (Coppini et al. 2001) making it suitable ingredients for cosmetic application.

Bedak sejuk is still prepared using traditional method and steps that is passed from generation to generation, which involved soaking of rice grains in water. The production of *bedak sejuk* is simple without involving any chemical reaction but it is time consuming as repeated soaking are needed to obtain fine rice flour at the end of the production after several months. During the soaking of rice grains, natural fermentation will occurs, which involves decomposition of rice starch by microorganisms in the soaking water (Dzulfakar et al. 2015).

According to Bloomberg (2019), natural cosmetics market will be worth around USD48 billion by 2025 due to high demand for natural health and wellness products and increasing awareness on harmful impact of synthetic chemicals. *Bedak sejuk* is a natural traditional beauty product with high potential to be expanded to business scale. It is usually produced in the form of loose powder or pearl shape droplets. To keep it up to date and modern, compact format of the powder need to be produced but there is no available study being executed in Malaysia. Thus, this research is emphasized on early investigation of the compact version of bedak sejuk using several of binding agent (alcohol and oil).

METHODOLOGY

RICE GRAIN SOAKING PROCESS

Local rice grain (Jasmine) were soaked in tap water at ratio of 1:1 (w/v) in a container with lid without washing and allowed to naturally ferment at ambient temperature (23 – 25°C) for 14 days. After 14 days, rice grains were filtered using a white cotton cloth. The remaining rice granules on the cloth were soaked again in tap water with ratio of 1:1. These procedures were repeated for five more times for a total soaking period of 84 days. At the end of the soaking process, the filtered rice granules *(bedak sejuk)* were washed and dried under sun.

PARTICLE SIZE DISTRIBUTION ANALYSIS OF BEDAK SEJUK

Bedak sejuk produced were divided into two groups: *bedak sejuk* without crushing and *bedak sejuk* crushed by using mortar and pestle. Both groups of *bedak sejuk* were sieved through 6 to 60 mesh Tyler screen. The weight of the *bedak sejuk* left on each sieve plate of different size was calculated by using the equation,

$$\text{Weight Percent}(\%) = \frac{\text{Weight of } \textit{bedak sejuk} \text{ left on sieve plate}(g)}{\text{Total weight of } \textit{bedak sejuk}\ (g)} \text{x}100\%$$

BINDING AGENT USED FOR THE PRODUCTION OF COMPACT POWDER

ALCOHOL

Bedak sejuk (10g) were weighed and prepared in 5 petri dishes. 10 ml of ethanol with concentration of 10%, 20%, 30%, 40% and 50% were prepared and mixed into the petri dished containing *bedak sejuk* respectively. The mixture was pressed to remove the trapped air bubbles inside and to form compact powder. The mixture was then dried at 30°C.

This experiment was repeated using isopropyl alcohol to replace ethanol.

POLYVINYL ALCOHOL

Samples of 50 ml polyvinyl alcohol solution were prepared respectively by mixing PVA and distilled water in different composition: 1wt%, 2wt% and 3wt%. The mixture was heated up to 90°C while stirred using a magnetic stirrer to form homogeneous solution. 10 g of *bedak sejuk* sample was mixed with 7 ml of PVA solution in mortar and stirred with pestle. The mixture was pressed to form compact powder, and then dried at 30°C.

MAGNESIUM STEARATE AND JOJOBA OIL

Three samples of 10 g powder mixtures were prepared respectively with different composition: 1wt%, 2wt% and 3wt% (e.g. 9 g *bedak sejuk* and 1 g magnesium stearate). Some drops of jojoba oil were added into the powder mixture, stirred for well-mixed and pressed to form compact powder. The compact powder was dried at 100°C in the oven, and then cooled at room temperature.

CHARACTERISTICS OF COMPACT BEDAK SEJUK

FTIR ANALYSIS

Chemical bonds and functional groups exist on *bedak sejuk* and compact bedak sejuk formed were detected by using FTIR (Brand: Thermo Scientific). Wavenumber of 400 – 4000 cm^{-1} was used as the base line. A very small amount of sample (1g) was prepared and placed under the sensor. The sensor is lowered down to touch the sample for analysis. When infrared radiation was emitted, some infrared rays were absorbed by the *bedak sejuk*. The infrared rays penetrated out of the sample were detected by the FTIR detector as spectrum. The result was shown on the software installed in the computer which is connected to the FTIR machine.

DROP TEST

The cracking of solid compact bedak sejuk produces was tested by dropping it from a height of 1m.

RESULT AND DISCUSSION

RICE GRAIN SOAKING PROCESS

For the production of cooling powder, a ratio of 1:1 with 250g of local rice was soaked in tap water in closed container at ambient temperature (23 - 25°C). This process was took place for 84 days with the changing of soaking water every 14 days. During soaking process, natural fermentation and rice decomposition occurred where microorganisms present use rice granules as a carbon source for growth (Yih 2015). Throughout this process, the size of the rice grain decreased. Additionally, unpleasant odor was produced due to the production of hydrogen sulphides (Linderholm et al. 2008). At the end, rice pastilles were filtered by using a muslin cloth, rinsed with water and dried under the sun.

PARTICLE SIZE DISTRIBUTION ANALYSIS OF BEDAK SEJUK

The sample of bedak sejuk was divided into two groups, bedak sejuk without being crushed and bedak sejuk being crushed using a mortar and pestle. Figure 1 shows the observation of both groups of sample. Cumulative weight fraction by particle size of bedak sejuk was shown in Figure 2 and 3. More than 60% of the bedak sejuk without crushing were in size of 1000 - 2000μm. For crushed samples, finer bedak sejuk of sizes ranging from 150 - 250μm were obtained in which 50% of the samples were bigger than 150 μm. Finer samples is believed to facilitate easy molding powder into compact format and shape (Amidon et al. 2017; Kang, 2005).

A

B

FIGURE 1. Bedak sejuk without crushing (A) and Bedak sejuk after crushing (B)

FTIR ANALYSIS OF BEDAK SEJUK

From FTIR spectra shown in Figure 4, the important peaks of rice cooling powder which related to its physical and chemical characteristics were observed. Result of cooling powder will then be used as a control to be compared with compact bedak sejuk formed in the following experiment. The FTIR spectra shows the peaks at 3344.3 cm^{-1} and 2931.1 cm^{-1}, which represents the hydroxyl group (OH) and C-H stretching vibrations, respectively. Absorption peaks at 1651 cm^{-1} arises from the stretch of C=O bond. C-O-H band were exhibited at 1152 cm^{-1} and 1078.4 cm^{-1}, while C-O stretch was exhibited at 1017.5 cm^{-1}. The peaks observed in the region at 950 cm^{-1} to 750 cm^{-1} represents carbohydrate content (Amir et al 2013; Dzulfakar 2016; Said et al. 2015).

SUITABILITY AND CONCENTRATION OF BINDING AGENT USED FOR THE PRODUCTION OF COMPACT POWDER

ALCOHOL

Table 1 and 2 show the observation made on compact powder formed by using ethanol and isopropyl alcohol as binding agent, respectively. When alcohol solution was added to the cooling powder, it tended to act as wetting agent. The alcohol solution dispersed uniformly and filling the spaces between the powder particles by adhesive force. This adhesive force attracted the powder particles to stick together, in which this process is called wet granulation (Arndt et al. 2018; De Simone, 2018). The mixture of alcohol and cooling powder was dried at 30°C to remove the alcohol. As alcohol exhibits high volatility, it evaporated very quickly. This rapid evaporation brought to ‘tightening’ effect on cooling powder to form compact powder (Barrett-Hill 2015). However, there is no formation of chemical bond between alcohol and cooling powder. After drying process, no smell of alcohol could be detected from the compact powder formed.

For both alcohols, the graphs before and after drying process exhibit the same trend and similar transmittance as that of pure cooling powder. This indicates that the addition of alcohol does not bring any effect chemically and there is no formation of new chemical bond which could hold the cooling powder particles more tightly. Hence, the compact powders formed were brittle and easily break as proved by the drop test. As the alcohol was almost evaporated completely, the compact powders formed by the addition of different alcohol concentrations were broken into pieces. This means that concentration of alcohol brings no effect on hardness of compact powder formed. It can be concluded the alcohol acts as wetting agent but not binding agent. Even though the compact powder formed was brittle, the cooling powder could be applied easily onto the skin.

POLYVINYL ALCOHOL AS BINDING AGENT

Table 3 shows the observation made on compact powder formed by using polyvinyl alcohol as binding agent. During PVA solution preparation, the water solution of PVA powder exhibited stickiness as glue. When PVA solution was mixed with cooling powder, PVA solution hold the cooling powder tightly until the powder agglomerates to form hard, solid compact powder. After drying at 30 °C, it was observed that the surfaces of compact powders are smooth. There were only few fine crack lines on sample 1, but no crack line on sample 2 and 3. This result was caused by the interaction among the hydroxyl groups of PVA and starch content in cooling powder. PVA had high interest due to its biocompatibility, reactivity, nontoxicity and solubility in water, which made it similar to starch in cooling powder. PVA also contained hydroxyl groups (OH) which interacted to form hydrogen bonds. In addition, cooling powder exhibited nucleating ability that promoted crystallization of the vinyl polymer. At the same time, this resulted in an improvement of tensile strength of compact powder formed. Thus, the PVA compact powder was harder than compact powder formed by mixing with alcohol as permanent inter-molecular and intra-molecular bonding were formed by PVA with cooling powder (Woranuch et al. 2017). This affects the result of drop test. The higher the concentration of PVA solution, the stronger the binding force of PVA with cooling powder, the harder the compact powder formed to resist the impulsive force act on it. The highest concentration of PVA used in sample 3 makes it harder than sample 2 and 1.

Figure 5 shows the comparison of FTIR results between pure cooling powder and compact powder made from PVA solution. The trend for both graphs is similar but exhibits different transmittance. PVA compact powder shows characteristic peaks at 3383.9 cm^{-1} (OH stretching), 2931.1

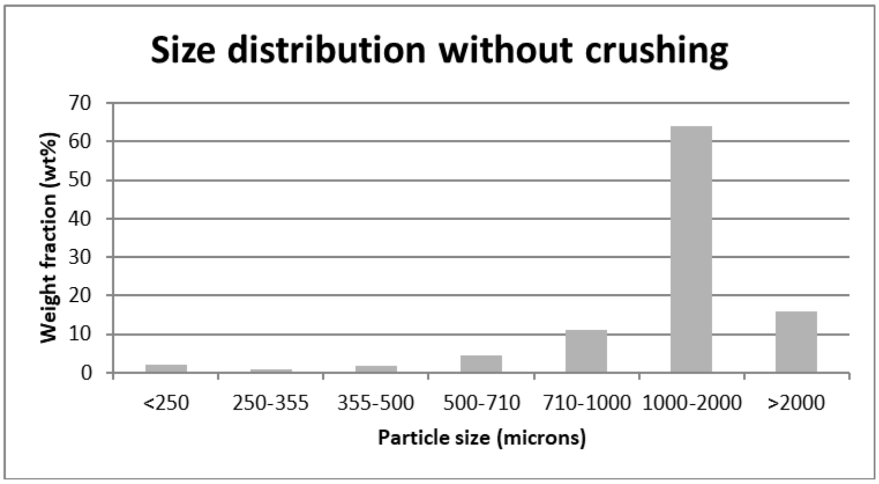

FIGURE 2. Size distribution of bedak sejuk without crushing

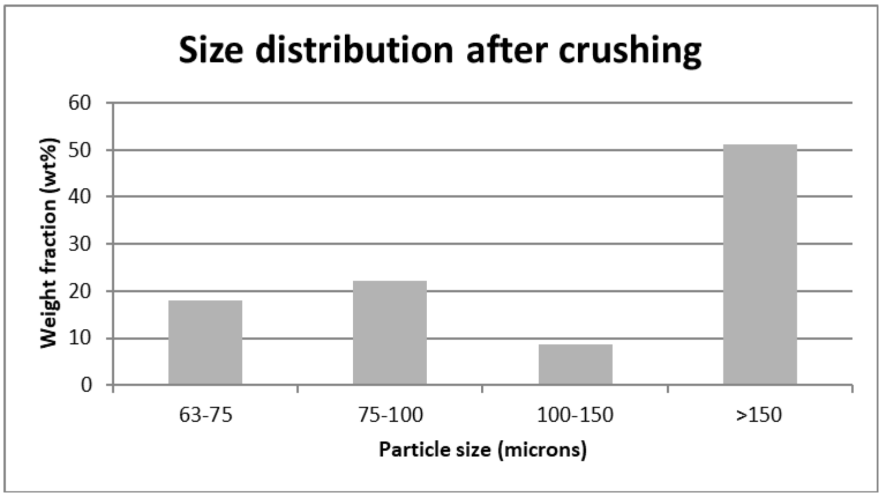

FIGURE 3. Size distribution of bedak sejuk after crushing

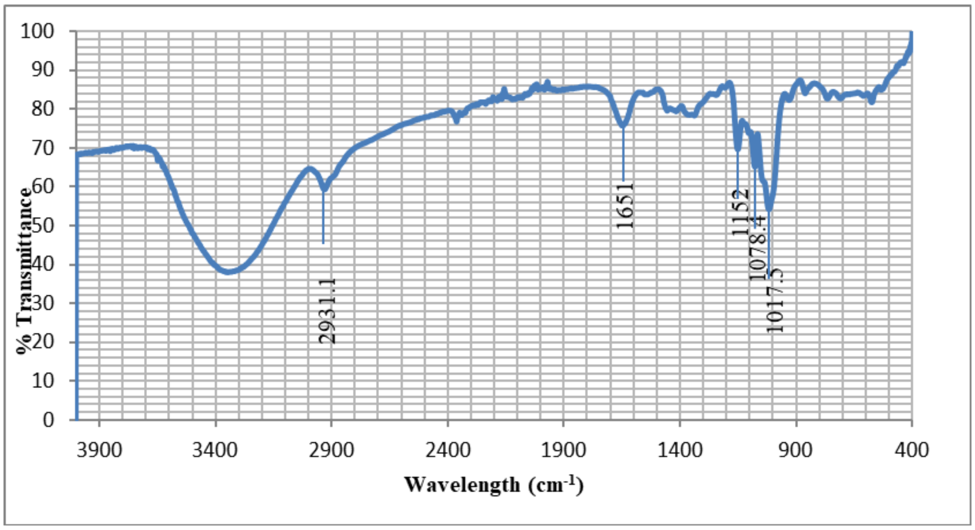

FIGURE 4. FTIR result of bedak sejuk

TABLE 1. Compact bedak sejuk formed by using ethanol

Concentration (%)	Observation	Note	Drop test	Note
10		++		++
20		++		+++
30		++		++++
40		-		++++
50		+++		++++

cm^{1} (C-H stretching), 1652 cm^{-1} (C=O stretching), 1153.3 cm^{-1} and 1080.2 cm^{-1} (C-OH stretching group) and 1023.1 cm^{-1} (C-O stretching). Cooling powder also exhibits the same functional group as PVA compact powder. However, it is noted that the OH peak of PVA compact powder was shifted to a lower transmittance compared with that of pure cooling powder. These shifts indicated that new hydrogen bonds formed in the compact powder were stronger than the existing hydrogen bonds in pure cooling powder (Woranuch et al. 2017).

Compact powder was successfully formed by using PVA as binder. However, due to the formation of strong hydrogen bonding, the cooling powder could not be applied easily onto the skin.

MAGNESIUM STEARATE AND JOJOBA OIL AS BINDING AGENT

Table 4 shows the observation made on compact bedak sejuk formed by using magnesium stearate and Jojoba oil as binding agent. In this experiment, magnesium stearate acts as dry binder while jojoba oil acts as liquid binder. The most important characteristics of magnesium stearate are their adhesive and waterproof properties. Magnesium powder more waterproof. It also makes the powder more apt to adhere to the skin (Hobbs et al. 2017). Moreover, it has lubricating properties thus preventing any ingredients from sticking to equipment during the compression of powder. Then, jojoba oil mixes the powder together to form compact powder. From observation, the higher the amount of magnesium stearate, the smoother the surface of the compact powder formed. This is due to the lubricant property of magnesium stearate. By carrying out drop test, the shapes of compact powders were remained unchanged. Jojoba oil was strong enough to hold the powder particles together in a solid form. However, this made the compact powder could not be arises and applied onto the skin.

Figure 6 shows the FTIR result of compact powder made from magnesium stearate and jojoba oil. By comparison, there is occurrence of bonds which is caused by magnesium stearate and jojoba oil. The compact powder shows different characteristic peaks from cooling powder at 2921.9 cm^{-1} and 2851.8 cm^{-1} (CH stretch), 1739.8 cm^{-1} (C=O ester group), 1637.4 cm^{-1} (C=C alkene), 1540.4 cm^{-1} (C=C aromatic) and 1465.9 cm^{-1} (CH_2 bending). These functional groups are appeared on the magnesium stearate and various types of fatty acids inside jojoba oil.

TABLE 2. Compact bedak sejuk formed by using isopropyl alcohol

Concentration (%)	Observation	Note	Drop test	Note
10		++		++++
20		-		++++
30		-		++++
40		-		++++
50		-		+++

Parameter - = No cracking
+ = Cracking lines were observed
++ = Large size of broken pieces
+++ = Moderate size of broken pieces
++++ = Small size of broken pieces

TABLE 3. Compact bedak sejuk formed by using polyvinyl alcohol

Concentration (%)	Observation	Note	Drop test	Note
1		-		+
2		-		+
3		-		-

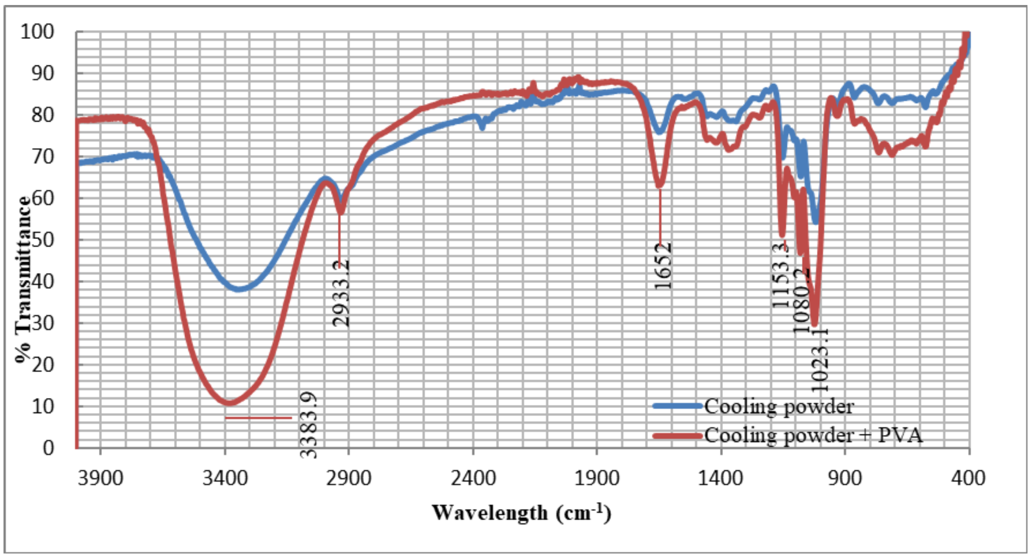

FIGURE 5. FTIR result of compact bedak sejuk with PVA

TABLE 4. Compact bedak sejuk formed by using magnesium stearate and Jojoba oil

Weight percentage (%wt)	Observation	Notes	*Drop test*	Notes
1		-		No change
2		Small cracked and oily surface		No change
3		Small cracked and oil surface		No change

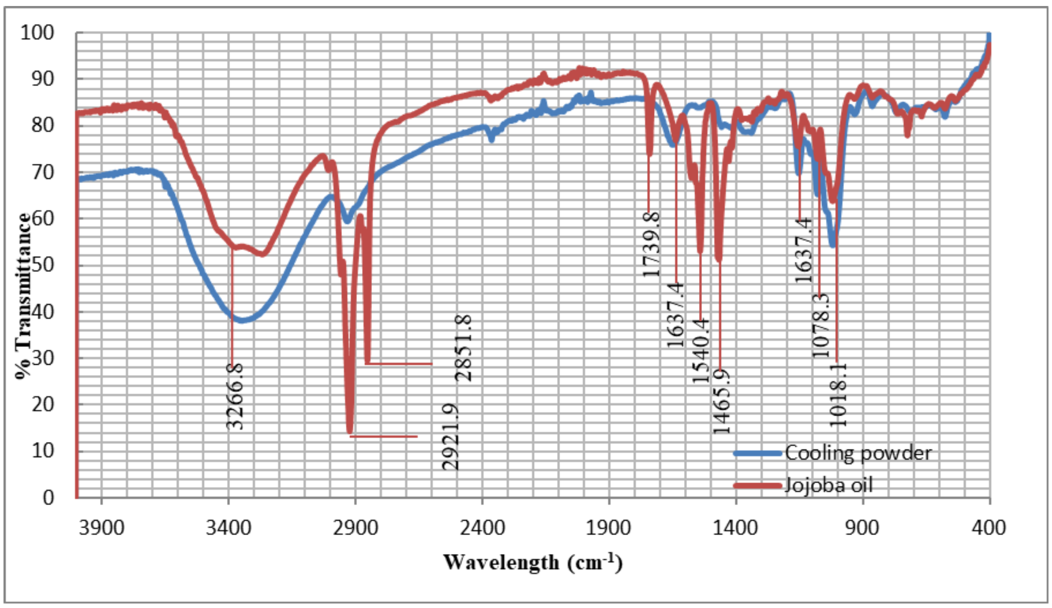

FIGURE 6 FTIR result of compact bedak sejuk with magnesium stearate and Jojoba oil

CONCLUSION

The particle size of bedak sejuk without crushing was found to be at the range of 1000-2000µm, while forcrushed samples, the particle sizes expected to become smaller in the range of 150-250µm. FTIR analysis shows that the functional groups exist on the bedak sejuk are hydroxyl (OH) bond, CH stretching, C=O stretching, C-O-H stretching and C-O stretching. Each type of compact powder formed by using different type of binding agent respectively showed their strengths and weaknesses. By taking easiness of applicability of powder onto the skin as the important criteria, alcohol (ethanol and isopropyl alcohol) are the best binding agent used to produce compact powder. But if structure of the compact format is considered, using polyvinyl alcohol, magnesium stearate and jojoba oil found to be strengthen the structure. Since compact powder format requires strength and easy application, future study on optimization of concentrations used for these products will help suggesting the best combination.

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DECLARATION OF COMPETING INTEREST

None.

REFERENCE

Amidon, G.E., Secreast, P.J. and Mudie, D. 2017. *Particle, Powder and Compact Characterization in Developing Solid Oral Dosage Forms*. London: Academic Press.

Amir, R. M., Anjum, F. M., Khan, M.I., Khan, M.R., Pasha, I. and Nadeem, M. 2013. Application of Fourier transform infrared (FTIR) Spectroscopy for the Identification of Wheat Varieties. *Journal of Food Science Technology* 50(5): 1018–1023.

Anon. 2010. Nilai bedak sejuk, *KOSMO!*

Arndt, O.R., Baggio, R., Adam, A.K., harting, J., Frenceschinis, E. and Klinebuddle, P. 2019. Impact of different drt and wet granulation techniques on granule and tablet properties : A comparative study. *Journal of Pharmaceutical Sciences* 107(12): 3143-3152

Barrett-Hill, F. n.d. Alcohol in cosmetic formulations, *beautymag online*.

Bloomberg. 2019. Business – Natural cosmetics market worth USD48.04 billion by 2025, *PR Newswire* (June 11, 2019)

Coppini, D., Paganizzi, P., Santi, P. & Ghirardini, A. 2001. Capacitá protettiva nei confronti delleradiazioni solari di derivati di origine vegetable. *Cosmetic News* 136: 15–20

Cosmeticinfo.org. 2016b. Isopropyl Alcohol. *Cosmetics Info*.

Cosmeticinfo.org. 2016c. Polyvinyl Alcohol. *Cosmetics Info*.

De Paepe, K.; Hachem, J.P.; Vanpee, E.; Roseeuw, D. & Rogiers, V. 2002. Effect of rice starch as a bath additive on the barrier function of healthy but SLS-damaged skin and skin of atopic patients. *Acta Derm. Venereol*. 82: 184–186

De Simone, V., Cavvavo, D., Lamberti, G., d'Amore, M. and Barba, A.A. 2018. Wet granualation process: Phemomenological analysis and process parameters optimization. *Powder Technology* 340: 411-419

Dzulfakar, M. A. A. 2016. Kajian Fermentasi Rendaman Beras Semula Jadi Bagi Menghasilkan Bedak Sejuk dan Penciriannya Sebagai Produk Kosmetik. Master Thesis, Universiti Kebangsaan Malaysia.

Dzulfakar, M. A. A., Noorhisham, T. K. & Tasirin, S. M. 2015. An initial study on the influence of natural fermentation on particle size changes during the production of bedak sejuk 2. Preparation of bedak sejuk. *Journal of Engineering Science and Technology*. Special Issue 4: 71–78.

Dzulfakar, M. A. A., Kofli, N. T. & Tasirin, S. M. 2016. Physicochemical properties, heavy metal content and microbial limit test of bedak sejuk. *Indian Journal of Science and Technology* 9(21): 1-6

Hasniza, N.I. and Noorhisham, T.K. 2019. Development of filtration system for the collection of soaking water during bedak sejuk preparation. *Jurnal Kejuruteraan* SI 2(1): 85-90

Hobbs, C.A., Saigo, K., Koyanagi, M. and Hayasyi, S. 2017. Magnesium stearate, a widely-used food additive, exhibits a lack of in vitro and in vivo genotoxic potential. *Toxicology Reports* 4: 554-559.

Johar, S.F.M., Dzulafakar, M.A.A. and Noorhisham, T.K. 2018. Amino acid and fatty acid profillin of local fermented rice grains and its soaking water. *Malaysian Applied Biology* 42(2): 113-117

Kang, S.J. 2004. *Sintering*. Butterworth-Heinemann

Li, J and Wu, Y. 2014. Lubricants in pharmaceuticaal solid dosage forms. *Lubricants* 2: 21-43

Linderholm, A. L., Findleton, C. L., Kumar, G., Hong, Y. & Bisson, L. F. 2008. Identification of genes affecting hydrogen sulfide formation in Saccharomyces cerevisiae. *Applied and Environmental Microbiology* 74(5): 1418–1427.

Marie. 2016. How to Turn Loose Powders into Pressed Powders. *humblebee & me*. http://www.humblebeeandme.com/turn-loose-powders-pressed-powders/

Mercado, C. & Verdon, D. 1988. Cosmetic in form of Pressed Powder. USA Patent (WO/1988/000039)

Nampi, P.P., Kume, S., Hotta, Y., watari, K., Itoh, Toda, H. and Matsutani, A. 2011. The effect of polivnyl alcohol as a binder and stearic acid as an internal lubricant in the formation and subsequent sintering of pray dried alumina. *Ceramics Internationals* 37 (8): 3445-3450

Said, H. N., Harijino & Kusnadi, J. 2015. Influence of natural fermentation on the morphology and physicochemical properties of indonesian rice flour and their effect on rice paper. *International Journal of ChemTech Research* 7(4): 1951–1959.

Satoshi, I. S., Ikuyama, R., Fujisaki, Y. and Sugimoto, K.-I. .2010. Abstracts: The effect of rinse water obtained from the washing of rice (YU-SU-RU) as a hair treatment. *International Journal of Cosmetic Science* 32: 391–394

Sulaiman, K., Teknologi Bedak Sejuk in *Utusan Malaysia*. 2015, Utusan Malaysia Online: Kuala Lumpur, Malaysia.

Woranuch, S., Pangon, A., Puagsuntia, K., Subjalearndee, N. & Intasanta, V. 2017. System : Transformation from Powder to Electrospun Nano fi bers under Hydrogen-bonding induced viscosity, crystallinity and improved. *RSC Advances* 7: 19960–19966.

Yap, L. Y. 2012. Komitmen Uncle Jamin Penghasilan Produk Bedak Sejuk Asli Kekal. *Buletin Mutiara*, p. 1.

Yih, O. K. 2015. Kajian Populasi Bakteria Asid Laktik Dalam Air Rendaman Beras Sewaktu Penghasilan Bedak Sejuk. Final Year Project, Universiti Kebangsaan Malaysia.

Team WOM, O. 2016. 10 Kelebihan Bedak Sejuk Untuk Putih dan Licinkan Muka. *Team WOM*. http://www.wom.my/kecantikan/kebaikan-bedak-sejuk/ [21 April 2016].