

Physicochemical Properties, Heavy Metal Content and Microbial Limit Test of *Bedak sejuk*

Mohd Alif Afife Dzulfakar, Noorhisham Tan Kofli* and Siti Masrinda Tasirin

Department of Chemical & Process Engineering, Faculty of Engineering and Built Environment,
Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor;
affedzulfakar@siswa.ukm.edu.my; ntk@ukm.edu.my; masrinda@ukm.edu.my

Abstract

Background/Objectives: At present, no study has been conducted to evaluate the safety of *bedak sejuk* as a cosmetic product. Hence in this study, the safety evaluation of *bedak sejuk* which is used daily by Malaysians was investigated. **Methods:** Five assessment tests were studied on physicochemical properties of *bedak sejuk* which includes determination of pH, moisture and volatile matter, matter insoluble in water, test for solubility of colours and fineness. Heavy metal content (lead) and microbial population limit tests were also executed. **Findings:** Results showed that *bedak sejuk* complies with the cosmetic powder standard, in which the pH of *bedak sejuk* was at 6.0 ± 0.3 , the moisture and volatile matter was at $1.1 \pm 0.6\%$, matter insoluble in boiling water was 68 ± 0.7 mg/mass, the results of colour solubility was rated colourless and presented in a normal particle distribution. For the heavy metal content, less than 3.5 ± 0.5 parts per million of lead in the sample was observed while for the microbial population limit test, was valued at 340 CFU/mL. **Application:** From these results, *bedak sejuk* has the potential to be marketed as loose powder in canisters or molded or compressed into cakes and inserted into mirrored compact cases, typically with an applicator, usually a pad, a puff or other implement.

Keywords: Bedak sejuk, Cosmetic Powder, Natural Cosmetic, Rice Starch, Safety Evaluation

1. Introduction

Cosmetic products are used every day by millions of consumers all around the world. There are varieties of cosmetic products in the market with various formulation types especially for skin care products. Physical attractiveness is perceived higher after makeup and with a higher likability of the body image, it brings a positive self-perception^{1,2}.

In Asia, skin whitening products are the most in demand cosmetic products at the present time^{3,4}. Even though skin whitening products normally applied on a small or limited skin area (face or neck), the whole body surface could be exposed too to the products as well⁵. Skin contact with substances in the cosmetic products will always involve some degree of risk. These products can cause consumer such as irritation, sensitization or photoreactions that can be produced by either natural or synthetic substances in the cosmetic products⁶. Therefore,

local effects were the primary safety concern of the cosmetic products.

In Malaysia, cosmetic products are regulated under the Control of Drugs and Cosmetics Regulation 1984 which is under the National Pharmaceutical Control Bureau (NPCB)⁷. In 2007, the regulations have been amended according to the ASEAN Cosmetics Directive (ACD)⁸ into the Guidelines for Control of Cosmetics Products in Malaysia (revision 02)⁹. As stated in the guidelines, any cosmetic products present in the market must be safe for the consumer; means do not cause any damage or harm to human health when applied under normal or reasonably foreseeable conditions of use. This guidelines are actually adopted and altered from the EU Directives¹⁰ and named as ASEAN Guidelines for safety evaluation of cosmetic products (Annex 1, Part 6 of the ACD)¹¹. Based on the ASEAN Guidelines for safety evaluation of cosmetic products, the safety assessment of any cosmetic products clearly relates to the manner of use. The cosmetic safety

*Author for correspondence

must be fulfilled by the manufacturers whom placed their cosmetic products in the market. If they cannot give scientifically proof of their product safety, the product will not be allowed to be placed in the market.

Nowadays, the awareness of buying a halal cosmetic is increasing not only in Malaysia but all around the world especially the country that Muslim is the majority citizen¹². Generally, when the cosmetic products are claimed as a halal cosmetic products, those products must not contain porcine by-products and derivatives and alcohol. Besides, those products and any accessories that accompany them must be comply with Islamic or Shariah law¹³. In Malaysia, JAKIM or Islamic Development Department of Malaysia is the only certification body that responsible for granting the halal certification to cosmetic manufacturer. The halal certification will only issue if the cosmetic product comply with the Malaysian Standard MS 2200:2008 requirements which state that any cosmetic product must be safe and non-hazardous to consumer¹⁴.

The concept of halal cosmetic covers all aspects of the management system; the substances of the cosmetic product must be a halal ingredients, the manufacturing, storage, packaging and logistic are take into account too. All of these must comply with Malaysian Standard MS 2200:2008 plus as for the substances, formulation and finished products must comply with the requirements of NPCB.

Cosmetic products are usually a concoction of ingredients, however *bedak sejuk* is unique as it comprises of only fermented rice starch. To prepare *bedak sejuk*, rice grains are soaked for three months or more, until the rice grains were completely dissolved. Normally, the soaking water are changed every week or sometimes every two weeks to get rid of the unpleasant smell¹⁵. After that, the rice grains were filtered out and then soaked again in clean water. This step is repeated until the rice grains turns into a rice paste. To make the rice paste into pastilles, the rice paste was filled into a cone made from jackfruit leaf (*Artocarpus heterophyllus*) or banana leaf (*Musa* spp.) resulted in small orifice at the pointing end of the cone. Tiny droplets are dropped onto a large clean cloth and then all the pastilles will be placed under the sun to dry.

Normally these pastilles will be dried with pandan leaves or with local flowers such as jasmine (*Jasminum sambac*), magnolia (*Magnolia champaka*) and spanich cherry (*Mimusops elengi*) at a minimum of 3 days. As documented by Burhill (1966), in the past, *bedak sejuk*

was used unscented or scented and patted onto face and neck¹⁶. It helps to refresh the skin and gives clear complexion besides acting as a remedy for rashes and acnes^{17,18}. Usually, *bedak sejuk* is worn overnight to bed as a facial mask.

As for *bedak sejuk*, the typical safety assessments required by the NPCB are the microbiological and heavy metal test because *bedak sejuk* is actually a fermented rice starch product. Based on the amended final report on the safety assessment of *Oryza sativa* (rice) starch¹⁹, rice starch is safe to use. But no data is available for fermented rice starch safety. As for the halal certification, the small scale industry or homemade production rarely applied for the certification due to its small production sizes.

It is known however, that the fermentation process that occurs during the soaking of the rice grains could produce other compounds/substances such as alcohol or organic acids that may adhere on the rice paste. Thus, even though it is made with known ingredients and safe handling procedures, it may be necessary to check the properties of the *bedak sejuk* by appropriate testing. Hence in this study, *bedak sejuk* that is being produced in the laboratory which follows the traditional homemade methods were analysed based on the physicochemical properties, heavy metal (lead) content and microbiological test limit. The results from the evaluation should comply with Malaysian Standard MS 2200:2008 and the requirements of NPCB. These evaluations are important to determine the performance, quality and effectiveness of *bedak sejuk*.

2. Materials and Methods

2.1 Preparation of *Bedak sejuk*

Local polished rice grains variety was purchased from the local market. The soaking process follows the traditional methods of *bedak sejuk* production. In order to stimulate household conditions, the container used was not sterilized and tap water was used. 500 gram (g) of rice grains were soaked in tap water (w/v) and allowed to ferment naturally at ambient temperature for 14 days. After 14 days, the rice grains were filtered and then soaked again (w/v). The overall soaking process was 84 days or 6 times of soaking. Sample of *bedak sejuk* were collected at the end of soaking.

2.2 Assessments Test for Bedak sejuk

All tests were done based on the standard methods of test for skin powders by the Bureau of Indian Standards. The standard values for physicochemical properties of *bedak sejuk* are based on Bureau of Indian Standard IS 3959-2004 while the standard values for heavy metal and microbial population limit test are based on Guidelines for Control of Cosmetic Products in Malaysia (Revision 02) 2009.

2.2.1 Determination of pH of Aqueous Suspension

10 g of *bedak sejuk* was taken in a 150 mL beaker. Then, 90 mL of freshly boiled and cooled water was added. It was stirred well to make a thorough suspension. The pH was determined within 5 minutes using pH meter.

2.2.2 Determination of Moisture and Volatile Matter

5 g of *bedak sejuk* was weighed accurately and placed in a porcelain or glass dish. It was dried in an oven at a temperature of 105°C to a constant mass. The moisture and volatile matter of sample were calculated based on the equation below.

$$\text{Moisture and volatile matter (percent by mass)} = \frac{100 \times M_1}{M}$$

where,

M_1 = loss in mass in g on drying, and

M = mass in g of the material taken for the test.

2.2.3 Determination of Matter Insoluble in Boiling Water

1 g of *bedak sejuk* was weighed and transferred to a 500 mL beaker. Sample was moistened with a little rectified spirit. 200 mL of water was added into the beaker and boiled. It was allowed to settle and the supernatant was filtered through Gooch crucible. The residue was washed with water and transferred completely to the filter. The remaining residue in the crucible was dried at $105 \pm 2^\circ\text{C}$ to obtain a constant mass.

$$\text{Matter insoluble in boiling water (percent by mass)} = \frac{100 \times M_1}{M}$$

where,

M_1 = mass in g of the residue, and

M = mass in g of the material taken for the test.

2.2.4 Test for Solubility of Colors

50 mL of water was added into a beaker containing 1 g of *bedak sejuk*. Then, it was boiled for 15 minutes and filtered. From this filtered solution, 10 mL of sample was taken and 15 ml of rectified spirit was added. It was refluxed for 15 minutes and filtered. The filtrate should be colorless or faintly colored.

2.2.5 Determination of Fineness

About 10 g of *bedak sejuk* was placed in specified (standard sieve 150 micron) sieve. It was washed by means of slow stream of running tap water and finally with fine stream from a wash bottle until most material has passed through the sieve. The water should be completely drained from the sieve. The residue was carefully transferred to a tarred watch glass and dried at 105°C for constant mass. The fineness of all samples was calculated.

2.2.6 Test for Heavy Metal

2 g of *bedak sejuk* was added in a crucible and heated on a hot plate. Then it is heated in a muffle furnace at 600 °C to a constant mass. 3 mL of dilute hydrochloric acid is added, and the volume was made up to 100 mL once there was no dissolution. The solution was then filtered. 25 mL of the filtrate is transferred into a Nessler cylinder. In another Nessler cylinder, 2 mL of dilute acetic acid is added with 1 ml of standard lead solution and the volume was made up to 25 mL with water. 10 mL of hydrogen sulphide solution was added to each Nessler cylinder and the volume was made up to 50 mL with water. The solution was mixed and allowed to stand for 10 min. The colour produced in these two Nessler cylinders was compared.

2.2.7 Microbiological Analysis

Bedak sejuk was analysed for the determination of total bacterial count. The sample, serially diluted in physiological buffer and appropriate homogenized, was then spread on Tryptone Soya Agar (TSA; Oxoid) and incubated at 37°C. The plates were observed after 24 h and after 5 days and the number of colony-forming units (CFU/mL) was determined. The analysis was performed in duplicate.

All samples were measured at least in triplicate. The basic statistical method was used to calculate the means and standard deviation of the triplicate results of each sample.

3. Results and Discussion

Control of Cosmetic Products in Malaysia (Revision 02) 2009, *bedak sejuk* can be classified as make-up powders¹⁹. As mentioned previously, *bedak sejuk* is fermented rice based cosmetic, where it consists of only one material which is starch (fermentable) which needs to comply to the standard or requirement of all materials. In this study, the standard of the material used followed the Indian Standards that is adopted by the Bureau of Indian Standards²⁰.

Five tests were conducted for checking the physicochemical properties of *bedak sejuk*. While heavy metal test for lead and and microbiological limit test were conducted too. The values for each test were calculated using suitable formulae. The values were then compared with standard values specified from the Guidelines for Control of Cosmetic Products in Malaysia (Revision 02) 2009 for heavy metal content and microbial limit population. While the standard value for physicochemical properties of cosmetic powder are compared with standard value from Bureau of Indian Standard IS 3959-2004²⁰. Tables 1 and 2 shows the value of each test. These results are the first ever reported results for the safety evaluation of *bedak sejuk*.

Table 1. Physicochemical properties of *bedak sejuk* (Bureau of Indian Standard IS 3959-2004)

Characteristics	Standard value	Bedak sejuk's value
pH value	5.5 – 9.0	6.0±0.3
Moisture and volatile matter (% by mass)	2.0 (Maximum)	1.1±0.6
Matter insoluble in boiling water (mg)	90 (Maximum)	68±0.7
Solubility of colours	Colourless	Colourless
Fineness (Residue on 150 µm, % by mass)	0.5 (Maximum)	0.84±0.3

The results show that the evaluated *bedak sejuk*, complied with the specific standards by both Guidelines for Control of Cosmetic Products in Malaysia (Revision 02) 2009 and Bureau of Indian Standard IS 3959-2004. A cosmetic product is considered safe if it has a pH value in range of 5.0 to 6.5²¹. Choosing a product that is too high or too low in pH will affect skin either nourishing or irritating it^{22,23}.

From the result, pH value of *bedak sejuk* is 6.2 which is within the safe pH range for cosmetic. Whilst for all others physicochemical properties show values within the standard values. Face powders shall essentially be similar to body powder described which may contain small amounts of perfume and colouring matter except that it shall be of finer particle size and free from grit. From the solubility of colour test, *bedak sejuk* can be rated as colourless which complies the standard of face powder. But then, result for fineness beyond the maximum value of the standard. This shows that the rice grains need to soak for few more intervals to get more fine particles.

According to Guidelines for Control of Cosmetic Products in Malaysia (Revision 02, 2009), cosmetic product are safe if it is complying with the heavy metal and total microbial limits⁹. The heavy metal in cosmetic products such as lead should be below 20 parts per million (ppm) and the total microbial count limit should not higher than 1000 CFU/mL or CFU/g.

Table 2. Heavy metal content and microbial population limit test

Characteristics	Standard value	Bedak sejuk's value
Heavy metals (as Pb), parts per million (ppm), max	20	3.5±0.5
Microbial Population (CFU/mL)	<1000	340

*Standard value based on Guidelines for Control of Cosmetic Products in Malaysia (Revision 02, 2009)

The results for heavy metals were found to be less than 2 ppm which is lower than the standard value at 20 ppm. As *bedak sejuk* using tap water during its production, there are few metals like Fe, Cu, Zn, Pb and Mn in the tap water. But as for the cosmetic safety, Pb content is more concern compared to other metals. Pb is almost not detected in tap water in Malaysia². From the results, there are about 3.5 ppm of lead in *bedak sejuk*. This probably due contamination occurred during the fermentation process. But the value is too low to consider as dangerous for safety using. For the microbial limit population, the result was at 340 CFU/mL which complying the standard value. From the results, the level of microbiological contamination is basically caused by a non-sterile condition during the production of *bedak sejuk*. The value of microbiological contamination shows the quality

standards and operation control depends heavily on the artisan's skill and hygiene. This contamination value of *bedak sejuk* should be maintained during its use, in spite of the inescapable contamination by the users. Addition of preservative in the product can be done to control the microbial growth²³. But confirmation of the safety of *bedak sejuk*, toxicological tests need to be executed which include skin irritation or sensitization test.

4. Conclusion

The safety evaluation of *bedak sejuk* shows that it poses no harm, but more comprehensive assessments need to be done to claim that *bedak sejuk* is safe to be applied on skin. From these results, *bedak sejuk* has the potential to be marketed as loose powder in canisters or molded or compressed into cakes and inserted into mirrored compact cases, typically with an applicator, usually a pad, a puff or other implement. Based on this initial safety evaluation, *bedak sejuk* is complying with Malaysian Standard MS 2200:2008 and the requirements of NPCB.

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6. References

1. Ko SH, Lee CS. Structural relationship between self-esteem, appearance management behaviour, body image, interpersonal relationship and hope of adults workers. *Indian Journal of Science and Technology*. 2015 Apr; 8(8):783–89. DOI: 10.17485/ijst/2015/v8i8/69265.
2. Azlan A, Khoo HE, Idris MA, Ismail A, Razman MR. Evaluation of selected metal elements in commercial drinking water and tap water in peninsular Malaysia. *Jurnal Sains Kesihatan Malaysia*. 2011 Jan; 9(1):5–11.
3. Huixia Q, Xiaohui L, Chengda Y, Yanlu Z, Senee J, Laurent A, Bazin R, Flament F, Adam A, Piot B. Instrumental and clinical studies of the facial skin tone and pigmentation of Shanghai women. Changes induced by age and a cosmetic whitening product. *International Journal of Cosmetic Science*. 2012 Feb; 34(1):49–54.
4. Krishen AS, LaTour MS, Alishah EJ. Asian females in an advertising context: exploring skin tone tension. *Journal of Current Issues & Research in Advertising*. 2014 Feb; 35(1):71–85.
5. Desmidt B, Van Hoeck E, Rogiers V, Courselle P, De Beer JO, De Paepe K, Deconinck E. Characterization of suspended illegal skin whitening cosmetics. *Journal of Pharmaceutical and Biomedical Analysis*. 2014 Mar; 90:85–91.
6. Nohynek GJ, Antignac E, Re T, Toutain H. Safety assessment of personal care products/cosmetics and their ingredients. *Toxicology and Applied Pharmacology*. 2010 Mar; 243(2):239–59.
7. National Pharmaceutical and Control Bureau (NPCB). The Control of Drugs and Cosmetics Regulation 1984 (Amendment 2009). Ministry of Health Malaysia; 2009.
8. ASEAN. ASEAN Cosmetic Directives (ACD). Cosmetic Committee (ACC) heads of delegations meeting, ASEAN Secretariats, Jakarta: Indonesia; 2008 Mar.
9. MEI 2009. National Pharmaceutical and Control Bureau (NPCB). Guidelines for control of cosmetic products in Malaysia (Revision 02). Ministry of Health Malaysia; 2009.
10. EU. EU Cosmetic Directive 76/768/AAC. The European Union; 1976 Jul.
11. Kamaruzaman KA. Halal cosmetics: between real concern and plain ignorance. *The Halal Journal*. 2008 Mar–Apr; 3(4):26–28.
12. Hashim P, Mat Hashim D. A review of cosmetic and personal care products: Halal perspective and detection of ingredient. *Pertanika Journal Science and Technology*. 2013 Jul; 21(2):281–92.
13. MS 2200. Department of standard Malaysia. Malaysian Standard Part 1: 2008 Islamic Consumer Goods-part 1: Cosmetic and personal care- General Guidelines (p.16). Shah Alam, Malaysia: Department of Standard Malaysia. Printing Department, SIRIM Berhad; 2008.
14. Dzulfakar MAA, Tan Kofli N, Tasirin SM. An initial study on the influence of natural fermentation on particle size changes during the production of *bedak sejuk*. *Journal of Engineering Science and Technology*. 2015 Jan; 10(S4):71–8.
15. Burkill IH. A dictionary of the economic products of Malay Peninsula. 2nd Volumes. Ministry of Agriculture and Co-operatives: Kuala Lumpur; 1966.
16. Bodeker G. Health and beauty in Malay society. Health and beauty from the rainforest. Malaysian traditions of ramuan; 2009 Apr.
17. The secrets of traditional asian beauty. *Asian Scientist Magazine* [Internet]. [Cited 2015 Jul 29]. Available from: <http://www.asianscientist.com/2015/06/print/secrets-traditional-asian-beauty/June>.
18. Anon. Amended final report on the safety assessment of oryza sativa (rice) bran oil, oryza sativa (rice) germ oil, rice bran acid, oryza sativa (rice) bran wax, hydrogenated

- rice bran wax, oryza sativa (rice) bran extract, oryza sativa (rice) extract, oryza sativa (rice) germ powder, oryza sativa (rice) starch, oryza sativa (rice) bran, hydrolyzed rice bran extract, hydrolyzed rice bran protein, hydrolyzed rice extract, and hydrolyzed rice protein. *International Journal of Toxicology*. 2006;25(S2):91–120.
19. IS 15608. Bureau of Indian Standards. *Skin Powder – Specification (Second Revision)*; 2005.
 20. Takagi Y, Kaneda K, Miyaki M, Matsuo K, Kawada H, Hosokawa H. The long-term use of soap does not affect the pH-maintenance mechanism of human skin. *Skin Research and Technology*. 2015 May; 21(2):144–48.
 21. Schreml S, Szeimies RM, Karrer S, Heinlin J, Landthaler M, Babilas P. The impact of the pH value on skin integrity and cutaneous wound healing. *Journal of the European Academy of Dermatology and Venereology*. 2010 Apr; 24(4):373–78.
 22. Lambers H, Piessens S, Bloem A, Pronk H, Finkel P. Natural skin surface pH is on average below 5, which is beneficial for its resident flora. *International Journal of Cosmetic Science*. 2006 Oct;28(5):359–70.
 23. Campana R, Scesa C, Patrone V, Vittoria E, Baffone W. Microbiological study of cosmetic products during their use by consumers: health risk and efficacy of preservative systems. *Letters in Applied Microbiology*. 2006 Sep; 43(3):301–06.