Collagen face spray: facial moisturizer from chicken egg shell membrane to prevent premature aging

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Abstract. Skin is often exposed to various factors, so the premature aging process become one of the public’s concerns. One of the efforts to maintain skin beauty and prevent premature aging is to fulfill the intake of collagen in the skin. Collagen is a protein, which one is found at chicken egg shells membrane. So far, chicken egg shells are only seen as waste, pollute the environment and are not used properly. Therefore, this innovation to making face spray cosmetic products appears to be a new breakthrough in the utilization and waste treatment. It can function to moisturize and nourish the skin, soothe facial skin, anti-aging, relieves redness on sensitive skin and can give a smooth impression on makeup. The method used in this research is an experiment, the results based on anatomical pathology tests using microscopic 400x magnification with Sirius red staining obtained 52.8% collagen content in egg shell membrane, and pH test obtained value of pH 5.4 which meets the requirements for the safe value for the skin pH 4.5-6.5 and the results of the organoleptic test on collagen face spray product are odorless, colorless, and the material is in the form of a liquid face spray cosmetic.

1. Introduction
Skin is something that is clearly visible from a person, especially when interacting with other people, so skin aging is an unavoidable problem. The skin aging process is a dynamic process. The skin aging process causes histological changes in the skin layers. Skin aging is a biological process that occurs due to internal factors, namely genetics and DNA damage, external factors such as lack of nutrition, environmental conditions, exposure to free radicals, and so on that can affect appearance [1]. The skin aging process is reduced to a decrease in the physiological function of the skin which appears as a clinical manifestation of the skin which will continue with age. Therefore, successful aging is needed by every individual to build self-confidence by preventing skin aging [2]. In fact, with age, the formation of collagen in the skin will decrease, causing reduced skin elasticity and causing wrinkles on the face. One way to avoid premature aging is to provide collagen supplements on facial skin [3].

Currently, the desire of consumers to fulfill the need for cosmetic products made from natural and safe ingredients is increasing [4]. Consumers are increasingly active in choosing cosmetics made from natural ingredients with active functions on their skin and many of them even have the desire to pay more for cosmetics that promise and have more benefits for the skin [5]. Natural ingredients as a source of active substances that can be used to meet the needs of collagen in facial skin, one of which is chicken egg shell membrane. Chicken egg shell layer consists of a cuticle formed by more than 85% protein
vesicles. Eggshell membranes contain proteins, proteoglycans and glycosaminoglycans associated with type I, V and X collagen fibers. Collagen in eggshell membranes can produce hydrolyzed eggshell membranes which have become the main composition in cosmetic products to produce quality cosmetics that can prevent the formation of wrinkles on the skin [6]. Egg shells contain 2% water and 98% dry matter. The egg shell comprises 9-12% of the total weight of one egg, of which 94% is calcium carbonate. In addition, egg shells also contain fat, magnesium, phosphorus, potassium and sodium [7].

The egg industry has grown rapidly because people have started to raise awareness of nutrition by consuming eggs as a source of protein. However, as a result of this, there is a buildup of eggshell waste which causes environmental pollution [8]. The Environmental Protection Agency claims that eggshell waste is ranked 15th as a major food industry pollution problem. This is because if the disposal of eggshell waste is not carried out properly, it will cause the growth of fungi on the eggshell which can cause health hazards [9]. Based on these problems, we are interested in utilizing eggshell waste into a useful and economical product, considering that eggshell waste is easy to obtain and has not been widely used in beauty products.

In previous studies, eggshell waste is often processed into plant fertilizer because it can enrich the soil with calcium and maintain the pH level of the soil [8]. However, the utilization of eggshell waste in beauty products is still very minimal. Therefore, we are interested in innovating chicken egg shells by taking part of the membrane as a face spray product with an unprecedented collagen content. Face spray is currently in demand by the public because in addition to having a spray form that is easy to absorb so it is practical to use, face spray also helps keep facial skin fresh all day long. In addition, face spray tends to be used for all skin types and can be used anytime and anywhere. Face spray is a liquid that contains nutrients to restore moisture to facial skin. Face spray also functions to refresh facial skin and help soothe facial skin [10].

Collagen Face Spray which is made from eggshell membrane contains collagen which is suitable for teenagers to adults who have skin aging problems so that it can be used as an anti-aging agent. In addition, this product is also able to moisturize and nourish the skin, especially dry skin types, soothe facial skin, relieve redness of sensitive skin and help give a smooth impression on makeup. This Collagen Face Spray product is mainly made from eggshell membrane extract so it is safer to use at a more affordable price compared to other face spray products. This Collagen Face Spray product from eggshell membrane is expected to be a solution for Indonesian people, especially for those who have skin aging problems and dry skin, to be able to overcome these problems. This Collagen Face Spray innovation from eggshell membrane can open very promising business opportunities in the economic sector because in this modern era people are very concerned about skin health.

2. Materials and Methods
The research was conducted using an experimental method which aims to determine the best formula for face spray based on eggshell membrane based on the ratio of glycerin concentration to the physical quality of face spray preparations.

2.1. Materials
The tools used in this study include digital scales, filtration apparatus, 100 ml glass beaker, 3000 ml glass beaker, Erlenmeyer, test tube, vacuum rotary evaporator, extraction apparatus, homogenizer tube, electric microscope. The materials used include egg shells obtained from the community around Universitas Negeri Semarang, ethanol, glycerin, PVP, aquadest.

2.2 Methods
2.2.1. Separation of the egg membrane from the shell
The sample is in the form of egg shell waste as many as 300 pieces taken from the community around Semarang State University, the collected samples are then washed with water until clean and not sticky and then dried by drying. After drying, the chicken egg shell membrane can be directly peeled off and removed from the shell.
2.2.2. Eggshell membrane extraction
The eggshell membrane that has been separated is then weighed and put into the homogenizer tube. In the homogenizer tube, the extraction process was carried out using the macerization technique within a span of 24 hours using a solvent in the form of 50% ethanol with a ratio of 1:5. The filtrate that has been produced from the extraction process is put into the evaporator machine at a temperature of 50°C. The filtrate that has passed evaporation then enters the filtration machine. Furthermore, the filtrate enters the rotary evaporator machine until a thick filtrate is produced and the eggshell membrane extraction is ready to be used as the main ingredient in Collagen face spray products.

2.2.3. Identification of eggshell membrane collagen
Anatomical pathology test using 400x magnification microscope with Sirius Red staining. Because this test is judged to be able to show collagen fibers, this test is carried out by grading the density of collagen fibers in the histological preparations of the tested tissue from each preparation [11].

2.2.4. Identification of the Eligibility of Collagen Face Spray Product
Several Collagen face spray product formulas that have been made are then re-identified through several tests to ensure the best formula to be used and to test the feasibility of using the product. Several tests were carried out, namely pH test, organoleptic test, and humidity test.

2.2.5. Production collagen face spray
The first stage is the extract of the chicken egg shell membrane with a predetermined formula put into a beaker glass then added glycerin and PVP which has been dissolved in warm water, then stirred until homogeneous and then transferred into a spray bottle that has been prepared, the final stage is adding aquadest until the solution is 100 mL.

3. Results and Discussion

3.1. Results of Identification of Collagen levels in egg shell membrane
Based on the identification of collagen levels that have been carried out at the Anatomical Pathology Laboratory using a 400x magnification microscope with Sirius red staining, the results show that there is a collagen content of 52.8% in the chicken egg shell membrane which can be seen in Figure 1. The collagen fibers formed based on this identification are very many and very close to each other. In each Collagen Face Spray formula there are 30 mL of chicken eggshell membrane extract in a 100 mL bottle and 18 mL of eggshell membrane extract in a 60 mL bottle. The face requires a minimum of 1 mL of collagen in a day, so the collagen levels in the face spray can be said to have met the range of collagen needs for the skin.

![Figure 1. PA Test Results for Eggshell Membrane Collagen](image-url)
3.2. pH Test Result

Table 1. pH test results for collagen face spray formula

| Material               | Formula (%) | F1 | F2 | F3 |
|------------------------|-------------|----|----|----|
| Egg Shell Membrane     | 30          | 30 | 30 |
| Extract                |             |    |    |    |
| Glycerin               | 20          | 25 | 30 |
| PVP                    | 5           | 5  | 5  |
| Aquadest               | 45          | 40 | 35 |
| pH results test        | 4.3         | 5.4| 8.4|

Table 1 shows the test results for collagen face spray formula. Based on the pH test that has been carried out using a tool in the form of a pH meter that has been carried out on several formulas, namely F1, F2 and F3, it is concluded that the pH results obtained vary. In F1 it produces a pH of 4.3 which means that F1 is smaller (too acidic), while at F3 it produces a pH of 8.4 which means that F3 is larger (too alkaline) to meet the requirements of a pH value that is safe for the skin. The most suitable pH value was obtained from F2 with a pH value of 5.4 because the pH range at F2 already met the requirements for a safe pH value for the skin, namely pH 4.5 - 6.5.

3.3. Organoleptic test results of collagen face spray product

Table 2. Organoleptic Test Results

| Organoleptic Parameters | Organoleptic Observations | F1 | F2 | F3 |
|-------------------------|----------------------------|----|----|----|
| Color                   | Colorless                  | Colorless | Colorless | Colorless |
| Smell                   | Odorless                   | Odorless | Odorless | Odorless |
| Form of Substance       | Liquid                     | Liquid | Liquid | Liquid |
| Homogeneity             | Homogeneous                | Homogeneous | Homogeneous | Homogeneous |

The results of the organoleptic test observations did not show any significant changes in color, odor, or substance in the formula during storage, in other words the formula was still the same as when it was first made. Collagen Face Spray also meets the organoleptic requirements contained in SNI 16-4949-1998 regarding cosmetic spray products.

3.4. Results of Collagen Face Spray Moisture Observation in respondent

Table 3. Humidity Test Results

| Parameters | Time Span (minutes) | Before Use | 5 | 30 |
|------------|---------------------|------------|---|----|
| Water      | 29.5                | 56.0       | 56.7|
| Oil        | 13.2                | 25.2       | 25.5|

The moisture test was carried out on the selected formula, namely F2 using the Skin Moisture Meter and the results showed that the skin's moisture level increased with increasing time after using the product, of which 43-46% was good moisture vulnerability for the skin.
4. Conclusion
The eggshell membrane has a very high collagen content, which is 52.8% which was obtained based on the results of anatomical pathology tests using 400x magnification microscope with Sirius red staining. F2 is the most suitable and appropriate formula for use as a formulation in the manufacture of Collagen Face Spray cosmetic products because the resulting pH value of 5.4 meets the requirements for a safe pH value for facial skin which has a pH range of 4.5 - 6.5. The humidity produced based on observations on Collagen Face Spray products also increases with increasing time after using the product.

References
[1] Getoff N 2007 Radiat. Phys. Chem. 76 1577–86
[2] Ahmad Z and Damayanti 2018 Berkala Ilmu Kesehatan Kulit dan Kelamin – Periodical of Dermatology and Venereology 208–215
[3] Bolke L, Schlippe G, Gerb J and Voss W 2019 Nutrients 11 7–11
[4] Faria-Silva C, Ascenso A, Costa A M, Marto J, Carvalheiro M, Margarida H, Ribeiro, Simões S 2019 Trends Food Sci. Technol. 95 21–32
[5] Draelos Z D 2019 Dermatol. Clin. 37 107–115
[6] Cordeiro C M M and Hincke M T 2012 Recent Patents on Food, Nutrition & Agriculture 3 1–8
[7] Ray S, Barman A K, Roy P K and Singh K B 2017 The Pharma Innovation Journal 6 1–4
[8] Waheed M, Yousaf M, Shehzad A, Inam-Ur-Raheem M, Khan M K I, Khan M R, Ahmad N, Abdullah and Aadil R M 2020 Trends in Food Science and Technology 106 78–90
[9] Ajala E O, Eletta O A A, Ajala M A and Oyeniyi S K 2018 Engineering, Technology and Environment 14 26–40
[10] Rasheed N, Rahman S A and Hafsa S 2020 Research Journal of Pharmacy and Technology 13 1693–700
[11] Novitasari A I M, Indraswary R and Pratiwi R 2017 Odonto Dental Journal 4 13–20