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Stability Determination of the Various Cosmetic Formulations containing Glycolic Acid

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Abstract

Glycolic acid(GA) is well known the most effective cosmetic ingredient on the epidermal remodeling, accelerated desquamation and inhibitory effect on melanin synthesis. The various cosmetic formulations containing GA have not been reported in terms of stability. This study was to investigate the stability of three formulations(gel, cream, and ointment). The stability of obtained formulations was tested chemical and physical characteristics including the composition stability, hot-cool cycling, the variation of pH and viscosity, and the observation of color and odor. The experimental results showed that the gel and cream containing 5% GA, both formulations have proper stability in the centrifugal test, hot-cool cycling test, viscosity, pH stability and the observation of color and odor. On the other hand, the 5% GA ointment did not have stability. We concluded that the formulations of gel and cream are more suitable than ointment to use GA ingredient for developing cosmetic in terms of stability.

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I. Introduction

Glycolic acid (GA), chemical formula $C_2H_4O_3$ is the smallest compound of α-hydroxy acid (AHA). GA is used in the pharmaceutical industry as a skin care agent. The skin-care cosmetics containing GA have been shown an excellent capability to penetrate the skin, decrease acne, reduce stretch marks, decrease melanin pigmentation, and increase the efficacy of antioxidants (Couch & Howard 2002). Due to its wide range of activities, GA is one of the important cosmetic ingredients. Recently, previous studies have shown that widely conducted to learn more of the ingredient of excellent effect and for the possibilities of its use in pharmaceutical and cosmetic formulations (Dal'Belo, Gaspar, Berardo, & Campos, 2006), (Kornhauser,

Coelho, & Hearing, 2012), (Faria, Damasceno, & Ferrari, 2014). As an application of GA in the cosmetic preparation, there is a major problem that associated with essential consideration of the various formulation types and GA concentrations. The cosmetics may be the controlling factor in the release of the active ingredient from the formulation. The GA is water soluble, and the gel formulation is aqueous that contains no oils. Also, creams and lotions of oil-in-water emulsions are usually the preferred choice (Compos, Gaspar, Goncalves, Pereira, Semprini, & Lopes 2015). These group of formulas is either alcohol or water-based. While the ointment formulation is semi-solid, nearly water-free that is a greasy, sticky, emollient, protective, occlusive. This perspective, we consider which formulations can challenge the most stable and effective for GA ingredient.

The goal of this work was to investigate the stability of three different formulations. The stability tests were composed of measuring the chemical and physical characteristics as centrifugation, cycling, viscosity, pH, color, and odor at the triplicate temperature in a certain period of time. The stability determinations were analyzed on these cosmetic formulations in order to evaluate their usefulness to support GA. More to the immediate point, the stability of cosmetic formulation is key parameters for good quality product.

II. Material and Method

1. Material

The various formulations containing GA were prepared using the follow ingredients: Glycolic acid (Chemours, Wilmington, Delawere, U.S.), distilled water, glycerin, butylene glycol, hydroxyethylcellulose, methylparaben, disodium EDTA, cetearyl alcohol, polysorbate 60, isopropyl myristate, glyceryl stearate, stearic acid, phenoxyethanol, carbomer, triethanolamine, propylene glycol, mineral oil, petrolatum, sorbitan stearate and phenoxyethanol.

2. Preparation of the gel, cream, and ointment containing GA

The study was conducted on cosmetic formulations constituted by a gel, cream, and ointment containing GA. They were prepared in the laboratory according to the composition summarized in Table 1.

1) Preparation of a gel formulation containing GA (Sample A)

The gel base was made as follows; first, phase A was prepared by dissolving 1.3 g hydroxyethylcellulose in distilled water. This solution was heated at approximately 50° C and mixed using the Homomixer® (HM-U1.0, JPL Co, Seoul, Korea). Phase A was added to the mixture of 3 g glycerin, 3 g butylene glycol, 0.03 g disodium EDTA and 0.2 g methylparaben. The mixture was heated up until all of the ingredients melted at 50° C. Afterward, 5 g GA was added to the prepared gel base and mixed respectively. Then, the sample A (gel containing 5% GA) was stirred at room temperature until the mixture congealed.

2) Preparation of a cream formulation containing GA (Sample B)

The creams base was made as follows; 0.2 g carbomer was dispersed in distilled water (72.7 g). This solution (phase A) was heated at approximately 70° C and mixed using the magnetic stirrer (Hei-TORQUE recision 400, heidolph, Schwabach, Germany). Phase B was prepared by dissolving 2 g isopropyl myristate, 1.5 g stearic acid, 2 g glyceryl stearate, 3 g polysorbate 60 and 0.4 g phenoxyethanol in 8 g cetearyl alcohol and mixed at 70° C. 0.2 g Triethanolamine was dispersed in 5 g distilled water. These solutions (oil and aqueous phases) were heated at 70° C. Afterward, 5 g GA was added to the prepared cream base and mixed respectively. Homogenization of the aqueous phases with oil phases was achieved with the magnetic stirrer at an equal speed of 2,000 rpm for an appropriate time.

Table 1. The Chemical Composition of the Formulations (unit: g)

Samples	Composition	Quantity
Sample A (Gel)	Distilled water	87.47
	Hydroxyethylcellulose	1.30
	Glycerin	3.00
	Butylene Glycol	3.00
	Disodium EDTA	0.03
	Methylparaben	0.20
	Glycolic acid	5.00
Sample B (Cream)	Distilled water	72.70
	Carbomer	0.20
	Isopropyl myristate	2.00
	Stearic acid	1.50
	Glyceryl stearate	2.00
	Polysorbate 60	3.00
	Cetearyl alcohol	8.00
	Phenoxyethanol	0.40
	Distilled water	5.00
	Triethanolamine	0.20
	Glycolic acid	5.00
Sample C (Ointment)	Cetearyl alcohol	12.50
	Mineral oil	8.00
	Petrolatum	5.50
	sorbitan stearate	3.50
	Propylene glycol	13.50
	Glycolic acid	5.00
	Phenoxyethanol	0.40
	Distilled water	51.60

3) Preparation of an ointment formulation containing GA (Sample C)

The ointment base was made as follows; phase A was prepared by dissolving 8 g mineral oil, 5.5 g petrolatum, and 3.5 g sorbitan stearate in 12.5 g cetearyl alcohol. This solution was heated at 55° C and mixed using the Agi-mixer (Hei-TORQUE 100 value, Schwabach, Germany). The phase B was prepared to mix 13.5 g propylene glycol, 5 g glycolic acid, and 0.4 g glyceryl stearate using homo-mixer and heated at 55° C. Phase A and B were mixed and then 51.6 g warmed distilled water (45° C) added into the molten oil base (phase A+B) while stirring the whole mixture on a magnetic stirrer at an equal speed of 2,000 rpm for an appropriate time. The resulting ointment was carefully triturated in a mortar to avoid air bubbles in the formulation.

3. Determination of stability

The chemical and physical characteristics, including the centrifugation, hot-cool cycling, the variation of pH and viscosity, observation of color and odor were determined. We referred to previous study, John (1985) ‘

Fundamentals of stability testing' for construct the stability test manual.

1) Determination of stability using the centrifugal test
The composition stability of three formulations was measured using a centrifugation method (HA-1000-3 benchtop centrifuge; Hanil Science Medical, Daejeon, Korea). Test tubes were filled with 10 g samples in each and centrifuged at 1238 G for 30 min. If the samples remained homogeneous after 30 min, it was considered to have proper stability. The separation degrees are classified into 5 levels (level 5: No separation, Stable, level 4: 1 ㎜ separation, level 3: 2 ㎜ separation, level 2: 3 ㎜ separation, level 1: 4 ㎜ and more separation).

2) Determination of stability using the cycling test
The the cycling test was done by hot-cool cycling form 4 to 45℃, using a multi-room temperature humidity incubator (Multi-Room Temp. Humidity Incubator DS-114, Dawon Science, Korea). The samples were stored to cycle 24 hourly at each temperature (4℃, 25℃, and 45℃). Seven cycles were performed, and the appearances of the samples were observed at the end of each cycle. After cycling test, the stability of samples was measured by the appearance, color, and separation.

3) Viscosity measurement
The viscosities of the samples were measured under 2,400 ~ 3,600 cPs with the LV spindle 100 rpm, s64, at 2 mins. using a Brookfield viscometer (DV-E Viscometer, Brookfield, MA, U.S.A). The temperatures of measurement were at 4℃, 25℃, and 45℃ in triplicate and performed at each temperature for 4 consecutive weeks.

4) pH measurement
The pH measurement of the 3 different formulations containing GA was carried out with a pH-meter (Hanna instruments, USA). The pH of samples was monitored at 4℃, 25℃, and 45℃ at each temperature for 4 consecutive weeks. The measurements were performed in triplicate and the average value was determined.

5) Observation of coloration and odor
We observed the discoloration, odor and characteristic changes depending on the temperature (4℃, 25℃, and 45℃) for 28 days.

4. Statistical analysis

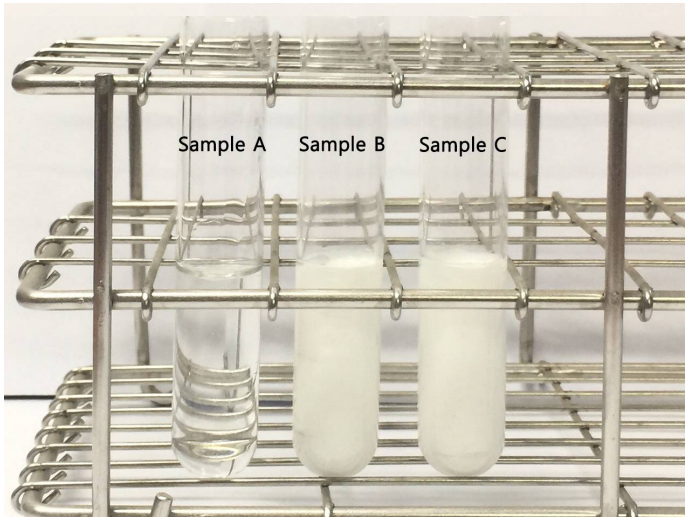
All statistical procedures were conducted using Spss ver. 24.0 software (SPSS Inc., Chicago, IL, USA). Date are presented as means and standard deviations. To compare the extent of stability, the paired t-test was used and the significance level was set at $P<.05$.

III. Results and Discussion

The samples were initially evaluated for physical stability using a centrifugation method, it is as of Figure 1. The sample A, B, and C have maintained homogeneity in centrifugation.

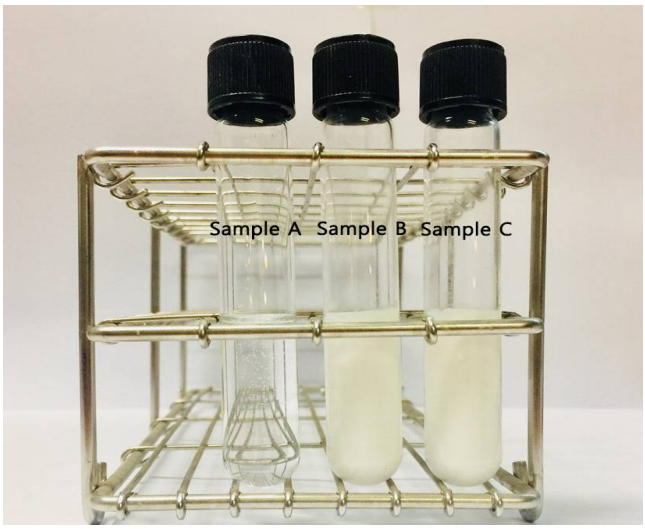
Cosmetic product in daily life may be exposed to extreme temperatures which may alter their properties. The investigation of the role of cool and hot cycling may put into evidence therefore not only a very important technological aspect but also an economic one: the environmental implication during transport and storage before selling of the product (Alina lliescu, 2005). The sample A, B, and C have passed three cycles of temperature testing from 4℃ to 25℃ and from 25℃ to 45℃. The cycling test results of prepared formulations were shown in Figure 2. All formulations, Sample A, B, and C was stable in hot-cool cycling.

The proper viscosity of cosmetics formulation improves spreadability and plays an important role in cosmetic product quality. As shown in Table 2, Sample B showed the highest viscosity and Sample C showed the lowest viscosity. The viscosity of sample A and B did not change during storage, in the condition of 4 weeks at 4℃, 25℃, and 45℃. In the previous studies, Ju(2017), the viscosity variation of the prepared gels have been little change, data showed that range of change was from 0.28% to 0.54% and it has presented that the cosmetic formulation is stable as the viscosity change has



Sample A : Gel containing glycolic acid 5%
Sample B : Cream containing glycolic acid 5%
Sample C : Ointment containing glycolic acid 5%

Figure 1. Centrifugation Test Result of the Prepared Formulations



Sample A : Gel containing glycolic acid 5%
Sample B : Cream containing glycolic acid 5%
Sample C : Ointment containing glycolic acid 5%

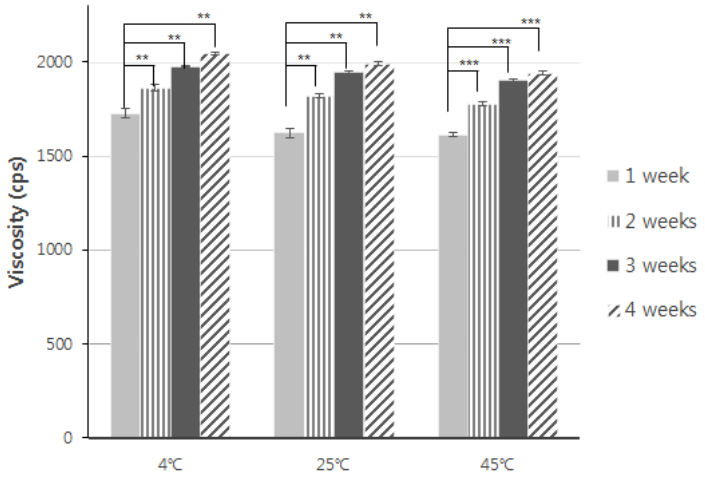
Figure 2. Cycling Test Result of the Prepared Formulations

little. In sample C, an increase of viscosity during storage was observed at 4℃, 25℃, and 45℃. Apparent viscosity value increased almost linearly from 1,600 cps to about 2,000 cps. The viscosity values were significantly higher after every week ($p<0.01$ and <0.001, Figure 3).

Table 2. Viscosity Variation of the Formulations (Unit : cps)

Temp.	Sample	1 week	2 weeks	3weeks	4weeks
4℃	Sample A	2995.00±5.00 (100%)	2998.33±2.88 (100.11%)	2996.00±5.29 (100.03%)	2997.00±1.73 (100.06%)
	Sample B	23505.00±5.00 (100%)	23501.00±1.73 (99.98%)	23505.00±5.00 (100%)	23501.00±1.73 (99.98%)
	Sample C	1726.66±25.16 (100%)	1863.33±15.27 **(107.91%)**	1976.66±5.77 **(114.47%)**	2046.66±5.77 **(118.53%)**
25℃	Sample A	2958.66±1.15 (100%)	2959.33±1.15 (100.02%)	2961.00±1.73 (100.07%)	2959.00±1.00 (100.01%)
	Sample B	23323.33±23.09 (100%)	23330.00±20.00 (100.02%)	23306.66±5.77 (99.92%)	23311.66±2.88 (99.94%)
	Sample C	1623.33±25.16 (100%)	1820.00±10.00 **(112.11%)**	1943.33±11.54 **(119.71%)**	1990.00±10.00 **(122.58%)**
45℃	Sample A	2589.66±0.57 (100%)	2589.00±1.15 (99.97%)	2589.33±1.15(10(9 9.98%)	2589.33±0.57 (99.98%)
	Sample B	23001.66±2.88 (100%)	23006.66±2.88 (100.02%)	23001.00±1.73 (99.99%)	23001.66±2.88 (100%)
	Sample C	1613.33±11.54 (100%)	1780.00±10.00 **(110.33%)**	1903.33±5.77 **(117.97%)**	1940.00±10.00 **(120.24%)**

Measurement data were done in triplicate, data shows mean±standard deviation
Sample A : Gel containing glycolic acid 5%
Sample B : Cream containing glycolic acid 5%
Sample C : Ointment containing glycolic acid 5%



Measurement data were done in triplicate, data show mean±standard deviation.
To compare the viscosity of sample C at 4℃, 25℃, and 45℃, by paired t-test.
Statistical significance was found at * : P<0.05, ** : P<0.01, *** : P<0.001.

Figure 3. Viscosity Value Variation of Sample C (Ointment)

Table 3. pH Variation of the Formulations

Temp.	sample	1 week	2 weeks	3weeks	4weeks
4℃	Sample A	3.82±0.02	3.86±0.01	3.84±0.00	3.81±0.01
	Sample B	3.85±0.01	3.87±0.02	3.86±0.01	3.85±0.02
	Sample C	3.82±0.06	3.81±0.05	3.73±0.00	3.74±0.00
25℃	Sample A	3.86±0.01	3.86±0.01	3.86±0.01	3.87±0.01
	Sample B	3.88±0.00	3.87±0.02	3.89±0.01	3.88±0.00
	Sample C	3.83±0.05	3.83±0.04	3.77±0.00	3.76±0.01
45℃	Sample A	3.87±0.02	3.89±0.01	3.87±0.02	3.89±0.00
	Sample B	3.91±0.01	3.92±0.02	3.91±0.01	3.91±0.01
	Sample C	3.86±0.05	3.84±0.04	3.84±0.01	3.86±0.01

Measurement data were done in triplicate, data shows mean±standard deviation
Sample A : Gel containing glycolic acid 5%
Sample B : Cream containing glycolic acid 5%
Sample C : Ointment containing glycolic acid 5%

Table 4. Stability of Discoloration and Odor for 4 Weeks

Sample \ Duration		1 week	2 weeks	3 weeks	4 weeks
Sample A	Odor	stable	stable	stable	stable
	Discoloration	transparency	transparency	transparency	transparency
Sample B	Odor	stable	stable	stable	stable
	Discoloration	transparency	transparency	transparency	transparency
Sample C	Odor	stable	stable	stable	stable
	Discoloration	transparency	transparency	transparency	transparency

Sample A : Gel containing glycolic acid 5%
Sample B : Cream containing glycolic acid 5%
Sample C : Ointment containing glycolic acid 5%

The pH of sample A, B, and C were determined at 4℃, 25℃, and 45℃ in 4 weeks (Table 3). The samples were analyzed in triplicate. The pH of sample A and B were 3.8 on average at all temperature during storage. The pH variation of the sample A and B have been little change, sample A and B which is gel and cream formulation, respectively showed an excellent stability.

The average pH of sample C is 3.8 at 4℃, 25℃, while the pH of sample C is 3.9 at 45℃. The pH of ointment has been changed a little by high temperature.

The CIR (Cosmetic Ingredient Review) panel of experts has reported that it is safe to use products containing GA above pH 3.5 based on previous studies (U.S FDA /CFSAN 2005). In this study, the pH of three typical formulations of a prepared cosmetic containing GA was on the average above 3.5.

There were no changes of coloration and odor in sample A, B, and C at 4℃, 25℃, and 45℃ in 4 weeks, it was shown in Table 4.

IV. Conclusion

The aim of this study is to analyze the stability of cosmetic which is three typical cosmetic formulations containing GA. The gel and cream formulations containing GA have exhibited stable chemical and physical characteristics, including composition (centrifugal test), hot-cool cycling, viscosity, pH, coloration, and odor.

The gel and cream formulations are practically the optimal choice to contain GA ingredient. GA is an acid and water soluble. The gel formulation is aqueous and creams (oil-in-water emulsion) is a higher water content which has contained no oils as well. While the GA ointment formulation has 13.5% oil contents. Although the additional oil phase components such as vaseline decreased the viscosity, The GA ointment formulation is unstable to increase of viscosity during storage at 4℃, 2 5℃, and 45℃.

References

Alina lliescu C. (2005). *Investigation on the thermal stability of technical and cosmetic emulsions.* (Unpublished Doctoral dissertation) RWTH Aachen University, Achen, Germany

Campos P., Gaspar L. R., Goncalves G., Pereira L., Semprini M., Lopes R. A. (2015) Comparative Effects of Retinoic Acid or Glycolic Acid Vehiculated in Different Topical Formulations. *BioMed Research International, 2015,* 1-6

Couch L. H., Howard P. C. (2002). Quantification of glycolic acid in cosmetic products using reversed phase high performance liquid chromatography. *Journal of Cosmetic Science, 24,* 89-95

Dal'Belo S. E., Gaspar L. R., Berardo P. M., Campos G. M. (2006). Moisturizing effect of cosmetic formulations containing Aloe vera extract in different concentrations assessed by skin bioengineering techniques. *Skin Research and Technology, 12*(4), 241-6

Ditre CM. (1998). Building your practice with glycolic acid peels. *Skin Aging, 6,* 48-53.

Fartasch M, Teal J, Menon GK. (1997). Mode of action of glycolic acid on human stratum corneum. *Archives of Dermatolgical Research, 289,* 404-409.

Faria W., Damasceno G., Ferrari M. (2014). Moisturizing effect of a cosmetic formulation containing pequi oil (Caryocar brasiliense) from the Brazilian cerrado biome. *Brazilian Journal of Pharmaceutical Sciences, 50*(1), 131-136

FDC legislation. (2011). Assurance of Cosmetics Among Europe. USA and Republic of Korea, *6*(1) 19-28

John, S. (1985). Fundamentals of stability testing. *International Journal of Cosmetic Science, 7,* 291-303.

Ju J. (2017). Effects of rhEGF and Palmitoyl pentapeptide-4 for skin regeneratin after glycolic acid peeling, (Unpublished Master's thesis). Wonkwang university, Iksan, Korea.

Kornhauser A., Coelho S. G., Hearing W. J. (2012). Effects of Cosmetic Formulations Containing Hydroxyacids on Sun-Exposed Skin: Current Applications and Future Developments. *Dermatology Research and Practice, 2012,* 1-6

Mukherjee S., Habif S., Rick D. (1999). Stable cosmetic conpositions with different pH emulsions. Partent No. US 5935589 A

Narayanan M., Sekar P., Pasupathi M., Mukhopadhyay T. (2017). Self-preserving personal care products. *International Journal of Cosmetic Science, 39*(3), 301-309

Olejnik A., Goscianska J., Zielnska A., Nowak I. (2015). Stability determination of the formulations containing hyaluronic acid. *International Journal of Cosmetic*

Science, *37*(4), 401-407

Ruiz M. A., Clares B., Morales M. E., Cazalla S., Gallardo V. (2007). Preparation and stability of cosmetic formulations with an anti-aging peptide. *Journal of Cosmetic Science*, *58*(2), 157-171

U.S. Food & Drug Administration. Labeling for Topically Applied Cosmetic products containing alpha hydroxy acids as ingredients (2005). Guidance for industry https://www.fda.gov/

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