

Low cost home-made turmeric (hydro) gel: Preparation, rheology and prediction of safe period for using

Abstract

Introduction: A single blind study was performed to find out the safe period for using the home made turmeric gels kept under normal condition on three types of customized formulations namely F1, F2 and F3. In this study, an economic preparation of turmeric gel with its safe period of use was proposed. F1 was formulated with preservatives (methyl paraben and propyl parabens in the ratio of 1:3), F2 was formulated with anti-oxidants (4 pieces of vitamin C tablets containing 250mg of ascorbic acid and 6 pieces of Vitamin E capsules each with potency 200IU). F3 was formulated without preservatives and anti-oxidants. In the study, samples (n=5 of each formulation) were investigated for organoleptic and physical properties including skin reactivity for 45 days by 15 volunteers.

Methodology: Our results showed that the preparations did not show any remarkable changes in color, after feel effect, grittiness and smoothness, stickiness, filming & spread ability along with wash ability within 30 days. pH of formulations remained the same over 45 days and pH of F2 was found below 5 and other 2 formulations are found 7.4. In the study, gel syneresis and gel swelling was investigated. Syneresis is exudation of fluid and swelling is taking up of adjacent liquid. Results showed both the phenomenon were absent for F1 and F2; but one sample of F3 showed gel swelling after 30 days although that sample did not show gel syneresis. Regarding skin reactivity, F2 and F3 did not show any reaction; but F1 did. One F1 sample showed skin reaction within 5 minutes after application. In current study microbial tests were carried out on F1, F2, F2 (fortified) and F3. Please note that for formulations F2 (fortified), the amount of each of the antioxidants was doubled. Microbial study results were expressed as 'cfu'/ml at a dilution of 10^5 .

Results and conclusion: Results are shown in table 4 and it shows for F1 'cfu' was too few to count up to a period of 21 days and beyond this period 'cfu' value became too many to count stating safe period of using of this formulation at home was 21 days. While for F2 the 'cfu' count was in a range of $(2.56-2.70) \times 10^8$ up to a period of 14 days and beyond this period count became too many to count stating safe period of using this formulation at home was 14 days. On the other hand, F2 (fortified) showed microbial count comparable to F1. For F2 (fortified) the 'cfu' count was $(1.75-1.84) \times 10^8$ up to a period of 21 days. Beyond this period count became too many to count. Therefore, safe period of domestic use for F2 (fortified) was 21 days. In the study, F3 preparation showed numerous microbial growth beyond two days stating safe period of use at home was only 2 days.

Keywords: turmeric, gel rheology, vitamin C, vitamin E, parabens, safe period of use

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Introduction

Herbal cosmetics are used mainly for beautification of body. These are the products where knowledge and experience of usage of herbs is blended with advanced technology so that safe and elegant beauty products can be formulated. From consumer's point of view, herbal cosmetics presently shows high demand and high turnover. For example, in India- the total cosmetic market is growing at the rate of 20-25% per annum and the herbal cosmetic industry shares almost Rs 200 crores out of an estimated Rs 2000 crores.¹ In UK, the selling margin of cosmetics was about 41% of the whole in 2014 constituting highest selling.² In Japan the production volume of cosmetic items is increasing tremendously. Here 164.7 thousand kilogram of lip balms were made in 2017 and the value was much higher than earlier.³ Considering personal expenses, it is reported that in USA about 31% of the total population spends 26-50 US dollars

per month.⁴ In Canada about 49% of the total population spends around 29 Canadian dollars per month.⁵ In this modern society herbal products undoubtedly have shown enormous popularity because herbs are safe and bio compatible. It is reported that even in areas where modern medicine is available, the interest on herbal medicines and their respective uses is continuing.⁶ Turmeric is a herbal item and the plant is known as *Curcuma longa* Linn, family: Zingiberaceae. This plant grows abundantly throughout Bangladesh, India and Pakistan. In Bangladesh it is extensively used as a spice in cooking and at present the safe use of Turmeric rhizome is well established.^{7,8} Apart from this use, it is popular in traditional medicine as well. For the last few decades, extensive works have been continuing to establish the cosmeceutical uses of this herb along with its pharmacological actions. In our society, turmeric based preparations are getting popularity and as such turmeric has been formulated in different forms like gel, cream, ointment and micro emulsion. Each form manifests its own

cosmetic values. Turmeric facial cream is one of the popular cosmetic preparation of turmeric since pre-historic era.^{9,10} Interestingly, although turmeric based cosmeceuticals are highly demanded, yet information regarding preparation of low cost facial turmeric (hydro) gel at home is still lacking. Moreover, safe period of usage of facial turmeric (hydro) gel kept under normal storage condition within the domestic premises has not been reported yet.

This present work therefore was undertaken to focus on preparing a low cost stable facial turmeric (hydro) gel with mandatory ingredients available at corner shop at home and at the same time to predict its period of safe use without showing any significant changes in its properties including the growth of microbes where storage conditions are normal.

Material and methods

Turmeric powder, sodium carboxymethyl cellulose (Na CMC), propylene glycol, glycerin, commercially available vitamin C tablets (Ceevit- 250mg of ascorbic acid, Square Pharmaceutical Ltd.), commercially available vitamin E capsules (E cap- 200 IU vitamin E, Drug International Ltd.), methyl paraben and propyl paraben (MERCK).

A) Preparation of (hydro)gel

The hydrogel (gel) was prepared following the process as described by Lloyd, 2005 where 6grams of gelling material was dispersed in plain distilled water¹¹. The dispersion was stirred gently to get a homogeneous, smooth and grittiness free mass¹¹. Following sonication for 5 minutes, the prepared gel was kept in an air tight container under normal condition until used.

B) Construction of rheogram

A certain amount of gel was taken on a glass slide covering an area of unit square cm. A cover slip was placed on it. Weights were added causing changes in areas. The process of adding weights [represented shearing stress (f)] was continued and respective changes in areas [representing rate of shear (ΔG)] were noted until the gel collapsed. The rheogram plotted as f versus ΔG indicated gel rheology whether the flow is 'Newtonian' and 'Non-Newtonian'.^{12,13,14}

C) Preparation of turmeric gel:

In the present study, turmeric gel was prepared in the above mentioned method with the exception of replacing water by turmeric extract.

D) Preparation of turmeric extract

i) Preparation of turmeric powder

2kgs of turmeric rhizomes were cut into pieces after cleaning and was sun dried followed by oven dried at 60°C for 3 days. The dried mass was then milled to powder. About 180g powder was got from 2kgs of turmeric rhizomes and was used for maceration.

ii) Soxhlet extraction

Turmeric powder (~170g) after sieving was taken in distill water (550ml) in a clean, dry soxhlet extractor. After 96 hours extraction-the solvent was removed, typically by means of a rotary evaporator, yielding the extracted compound. The non-soluble portion of the extracted solid remained in the thimble and the solvent (representing ~30 % turmeric extract) was collected. After that, the solvent was filtered repeatedly until the filtrate was clear and transparent.

E) Testing protocols for evaluating turmeric gel:

After completion, the prepared gel was subjected for property evaluations according the methods described by Arunrashed, Lovaleen prêt, Woo fong, Jannat 2019.¹⁵⁻¹⁸

1) Gel rheology

In order to investigate the flow pattern, several rheograms at different concentration levels were constructed. On the basis of rheogram pattern and exponential value, gel rheology was known.¹²

2) Organoleptic property

For property evaluation following tests were performed. The tests were done by 15 volunteers (male & female) of age between 18 to 22 years and prior to begin the test each of the volunteers was given a prepared questionnaire to fill. Later on the filled questionnaires were collected and analyzed. The following properties were given preferences for evaluation -

a) Appearance (color and clarity): The appearance of the gel was judged by its color and clarity.

b) After feel: Emollience and slipperiness was judged by feel.

c) Homogeneity and freedom from grittiness: The formulations were tested manually for grittiness (presence or absence of gritty particles and lumps) and smoothness.

d) Stickiness: This property was also checked manually.

e) Filming and spreadability: Ease of filming (smearing) and spreadability was judged by applying gel (1/2 tea spoonful) in a form of film on 1sq. inch area of forehead and time (minutes/seconds) taken for filming was noted.

f) Washing of film (washability): The ease of removal of smear formed on 1sq inch area of forehead using plain water was studied. Here time (minutes) taken and amount of water (in ml) used for complete removal of the film were recorded.

3) Physical property evaluation¹⁶⁻²²

Gel syneresis

This term is similar to bleeding that occurs in table jellies and gelatin desserts. It is reported that many gels often undergo contraction either spontaneously or slowly and exudes some fluid that gives rise to consequent squeezing out effect. This leads to continuous coarsening of the matrix or fibrous structure of gel causing hardening of its consistency. This phenomenon is called 'gel syneresis'. Following such 'gel syneresis,' the gel ultimately suffers a change in rheology and (thermodynamic) stability. Therefore 'gel syneresis' is an important property to investigate. Here the property was tested visually

Gel-swelling

Gel - swelling is the reverse phenomenon of 'gel syneresis'. In fact, a gelling agent is the basic material for making a gel. But the agent when comes in contact with a solvating liquid then an appreciable amount of the liquid may be taken up by the agent and the phenomenon may increase bulk volume of gel. This is 'gel swelling' which results due to 'gel-solvent' interaction. The more the interaction the less would be the strength of linkages that lies between the individual molecules of the agent causing a loss in structural integrity. In the present study 'gel swelling' was also checked visually.

4) Skin reactivity

After application the film of gel was checked for irritancy, redness, itching and swelling (if any) following a slightly modified approach as stated by Wilkinson and Moore.²²

Microbial test

Microbial growth was expected to grow in turmeric gel with time. Therefore, tests for microbial growth deemed important. Microbial test was carried out following a standard method using nutrient agar media.²³ In the study, serial dilution technique was used and results were expressed in terms of colony forming unit per ml ('cfu/ml'). Please note that a count greater than 300 was designated as 'too numeric to count' (TNTC) and a count less than 30 was designated as 'too few to count' (TFTC)

Results

Rheograms

Rheograms for Aloe vera, carbopol 940 and Na CMC at different concentrations are shown in Figure 1-3 respectively. Na CMC at 6% concentration produced a well-constructed rheogram (figure 3) and it was non linear with an exponent 1.34.

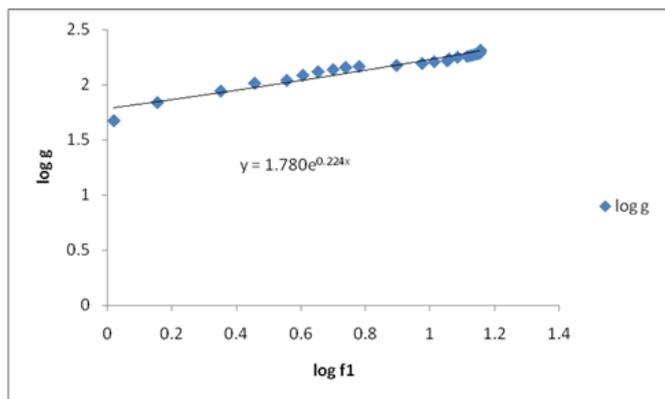


Figure 1 Rheogram for Aloe vera (hydrogel) (commercial variety).

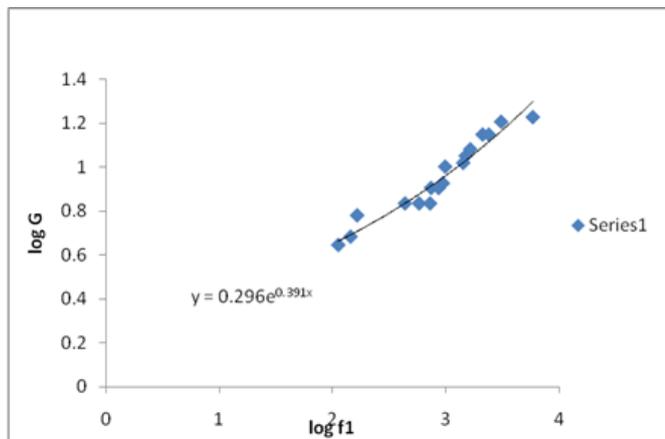


Figure 2 Rheogram for Carbopol 940 hydrogel (at 5% concentration level).

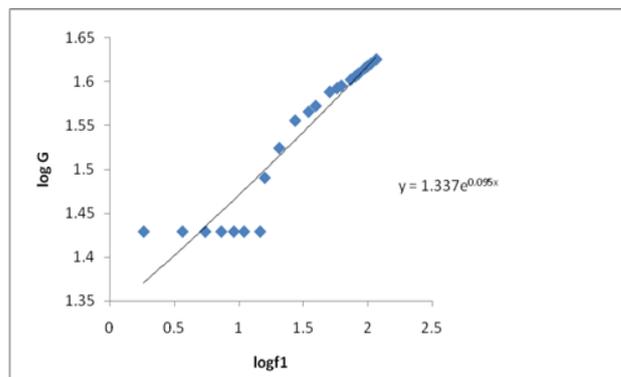


Figure 3 Rheogram for Na CMC hydrogel (at 6% concentration level).

Organoleptic, physical and skin reactivity test

In the study three types of formulations (F) namely F1, F2 and F3 were prepared using customized formulas where F1 represents formulation with preservatives only. F2 represents formulation with antioxidants only and F3 represents formulation without antioxidants and preservatives. In the study, the combination of parabens (1 part of methyl paraben and 3 parts of propyl paraben) was used as preservatives and commercially available. Vitamin C tablets (each containing 250 mg of ascorbic acid) and Vitamin E capsules (each having potency of 200 IU) were used as antioxidants. Stability test covering 45 days was carried out on them. During the tests, changes occurring in organoleptic properties and physical properties were monitored. For organoleptic evaluation, qualities given emphasis were appearance (color and clarity), after feel, grittiness and smoothness, stickiness, filming & spreadability and washability. For physical evaluation- pH, gel syneresis and gel swelling were given emphasis. In addition to organoleptic and physical property evaluation, skin reactivity test was performed also. In the study, tests were done on 5 samples of each formulation and results are shown in the following tables (table 1-3). Table 1 represents the results of organoleptic property tests. Tables 2 and 3 gives the results of physical and skin reactivity tests respectively.

Microbial tests

Microbial growth is a potential threat for product decomposition. It causes product spoilage and renders the product unusable for use. For water based preparations containing ingredient, chances of microbial growth are most likely to take place. Moreover, if any preparation contains any naturally occurring substance, then chances become many folds higher. Our preparation is a hydrogel and it contains Na CMC as a gelling agent. Therefore, microbial tests were mandatory to carry out. Results of the tests are shown in table 4. Please note that microbial study was done on F1, F2 F3 and F2 (fortified). Here F2 (fortified) was formulated with 8 piece of Vitamin C tablet (each containing 250mg of ascorbic acid) and 12 pieces of Vitamin E capsules (each having potency of 200IU) per 100g of gel.

Discussion

Turmeric skin preparations like turmeric facial cream, turmeric facial gel, turmeric whitening cream are popular and demanded since prehistoric era. Interestingly claiming on time period for safe use

of home-made turmeric hydrogel including selection of a suitable gelling agent along with the study of rheological behavior, working concentration and property evaluation is still lacking.

In our study, a number of gelling agents were tried (Figure 1-3); but 6% NaCMC was found the best for rheogram study and for turmeric hydrogel preparation. The rheogram of turmeric hydrogel was nonlinear and exponent of the rheogram was 1.34. Such results signifies that a continual addition of stress (f) on gel failed to produce a proportionate change in rate of shear (ΔG) (Figure 3). This means the relationship between ΔG versus f (values being taken on log scale) was not linear; rather nonlinear meaning Non newtonian. So the plot drawn with ΔG versus f was exponential and the exponent term was 1.34. As the exponent term was greater than unity so flow was Non Newtonian more specifically-pseudoplastic.¹⁴ Such results are in line with others. According to Martin et al, 1993 and Rawlins, 2004 polymers like cellulosic materials in solution exhibits pseudoplastic flow.^{12,14} Usually the rheogram for a pseudoplastic material begins at the origin (or at least approaches at which low rate of shear lies, where yield value is lacking). This makes the rheogram non-linear and at this point resistance of the material decreases with increasing rate of shear. Here the curved rheogram results from a shearing action on the long chain molecules of a linear polymer. As f increases it starts the normally disarranged molecules to align the long axes in the direction of flow causing a decrease in the internal resistance of material and allows a greater rate of shear at each successive shearing stress. Na CMC is a high molecular weight synthetic polymer of acrylic acid, cross linked with allyl sucrose and containing a high proportion of carboxyl group. In the present study it was used as a gelling material. Therefore, non linearity of the rheogram was in general agreement with others.

The present study was conducted for quality evaluation of the formulations spanning 45 days. Results showed that organoleptic properties of formulations (n=5 of each variety) did not change up to 30 days (Table 1). Beyond this time period samples of F3 formulations went little changes in color, stickiness, filming and washing. This may be ragded as a sign of product instability as these formulations were without preservatives and antioxidants.

Similar were the results with physical property tests. Here pH, syneresis and swelling occurring in F1, F2 and F3 were observed throughout 45 days. Results showed pH remained almost the same throughout (Table 2). pH of our skin lies on average below 5.¹⁹ It is slightly acidic and as such our skin can't tolerate a wide change in pH. A widely varied change in pH results skin irritation and discomfort¹². Therefore, our customized formulations were suitable for our skin.

Syneresis and Swelling are two degradative signs and symptoms of gels; hence these two properties were also evaluated. Results are shown in table 2. F1, F2 and F3 did not show signs of syneresis and swelling in samples except one. Here 1 sample of F3 showed swelling. In fact, hydrogels are preparations of gelling material in water. Here the material makes a cross linking with solvent molecule and makes a 3D network. Under unfavourable conditions, gel may release solvent molecule or it may further take up water molecule from outside. Here what actually happened needs exploration. In the study, skin reactivity of formulations was tested. All samples of F2 and F3 did not show any skin reaction except a single sample of F1 (Table 3). Such phenomenon can be explained on the fact that bio-chemical reactions exhibiting a bio response differs from person to person.

In the present study microbial tests were done on formulations. Results are shown in table 4. For F1 formulation, our results showed that microbial count was too few to count (TFTC) at 10^5 times dilution up to 21 days. Beyond this period, count became numerous. This result is comparable to F2 (fortified). Here count was in the range of $(1.75- 1.84) \times 10^8$ and like F1 count became numerous after 21 days. Thus for F1 and F2 (fortified) formulation, safe period of use was 21 days. On the other hand, F2 formulation in the present study failed to show similar results. Here results showed microbial count was measurable up to 14 days (Table 4). Beyond this time limit microbial growth became huge. Thus for F2 formulations the safe period of use was 14 days. For F3 formulation our results were frustrating as in the current study, F3 formulation showed enormous microbial growth after 2 days; thus the safe period of use for F3 was only 2 days. Here the count was 1.75×10^8 on 2nd day post preparation (Table 4). Such result was not unusual because this formulation was without preservatives and antioxidants.

Table 1 Organoleptic properties of F1, F2 and F3 homemade turmeric gels

Properties (n=5)	Day 1	Day 7	Day 14	Day 30	Day 45
Appearance (color and clarity)					
F 1	1. Acceptable	1.As before	1.As before	1.As before	1.As before
F 2	2. Acceptable	2.As before	2.As before	2.As before	2.As before
F 3	3. Acceptable	3.As before	3.As before	3. Slightly changed	3. Noticeably changed
After feel					
F 1	1. Cool	1.Cool	1.Cool		1. Coolness was less
F 2	2. Cool	2.Cool	2.Cool	1. Coolness was less	2. Coolness was less
F 3	3. Cool	3.Cool	3.Cool	2. Coolness was less	3. Coolness was less
Grittiness and smoothness					
F 1	1. Non gritty and smooth	1.As before	1.As before	1.As before	1.As before
F 2	2. Non gritty and smooth	2.As before	2.As before	2.As before	2.As before
F 3	3. Non gritty and smooth	3.As before	3.As before	3.As before	3.As before

Table continued

Properties (n=5)	Day 1	Day 7	Day 14	Day 30	Day 45
Stickiness					
F 1	1. Fairly sticky and soft	1.As before	1.As before	1.As before	1. Less soft and hard
F 2	2. Fairly sticky and soft	2.As before	2.As before	2.As before	2. Less soft and hard
F 3	3. Fairly sticky and soft	3.As before	3.As before	3.As before	3. Less soft and hard
Filming and spreadibility					
F 1	1.Easilyformed in 1 min	1.Easilyformed in 1 min	1.Easilyformed in 1 min	1.Easilyformed in 1 min	1. >1min was needed
F 2	2.As above	2.As above	2.As above	2.As above	2.As above
F 3	3.As above	3.As above	3.As above	3.As above	3.As above
Washability					
F 1	1. ~75 ml	1.~75 ml	1.As before	1.As before	1.~ 150 ml
F 2	2. ~75 ml	2.~75 ml	2.As before	2.As before	2.~ 150 ml
F 3	3. ~75 ml	3.~75 ml	3.As before	3.As before	3.~ 150 ml

Footnote: n,number of sample tested in each formulation; F1, Formulation with preservatives only; F2,Formulation with antioxidants (4 piece of vitamin C tablet and 6 pieces of vitamin E capsules) only; F3,Formulation without antioxidants and preservatives.

Table 2 Physical properties of F1, F2 and F3 homemade turmeric gels

Properties (n=5)	Day 1	Day 7	Day 14	Day 30	Day 45
pH					
F 1	1. 7.1–7.3	1. ND	1. ND	1. ND	1. 7.1–7.3
F 2	2. 4.8 - 5.1	2. ND	2. ND	2. ND	2. 4.8-5.1
F 3	3. 7.3–7.5	3. ND	3. ND	3. ND	3. 7.3–7.5
Gel syneresis					
F 1	1.NSA	1.NSA	1.NSA	1.NSA	1.NSA
F 2	2.NSA	2.NSA	2.NSA	2.NSA	2.NSA
F 3	3. NSA	3. NSA	3.NSA	3. NSA	3. NSA
Gel swelling					
F 1	1.NSA	1.NSA	1.NSA	1.NSA	1.NSA
F 2	2.NSA	2.NSA	2.NSA	2.NSA	2.NSA
F 3	3.NSA	3.NSA	3.NSA	3. NSA	3.Seen in I

Footnote: n, number of sample tested in each formulation; F1, Formulation with preservatives only; F2, Formulation with antioxidants (4 piece of vitamin C tablet and 6 pieces of vitamin E capsules) only; F3, Formulation without antioxidants and preservatives; ND, Not done; NSA, Not seen in any.

Table 3 Skin reactivity of F1, F2 and F3 homemade turmeric gels

Properties (n=5)	Day 1	Day 7	Day 14	Day 30
Skin reactivity				
F 1	1. Non-reactive in 90% of total participants			
F 2	2. NSA	2. NSA	2.As before	2.As before
F 3	3. NSA	3. NSA	3.As before	3.As before

Footnote: n, number of sample tested in each formulation; F1, Formulation with preservatives only; F2, Formulation with antioxidants (4 pieces of vitamin C tablet and 6 pieces of vitamin E capsule) only; F3, Formulation with no antioxidants and with no preservatives; NSA, Not seen in any.

Table 4 Microbial counts of F1, F2, F2(fortified) and F3 homemade turmeric gels

Days	Microbial count at (cfu/ml) at dilution of 10 ⁵			
	F1	F2	F2 (fortified)	F3
00	TFTC	(9.9X10 ⁷) -(1.05X10 ⁸)	9.5X10 ⁷	(3.5X10 ⁷)
02	TFTC	ND	ND	(1.75X10 ⁸)
07	TFTC	1.47-1.6X10 ⁸	1.27X10 ⁸	TNTC
14	TFTC	2.56-2.7X10 ⁸	1.45X10 ⁸	TNTC
21	TFTC	TNTC	(1.75-1.84)X10 ⁸	TNTC
30	TNTC	TNTC	TNTC	TNTC

NB: F1, signifies formulations containing preservatives only; F2, made up with antioxidant mixture of 4 piece of Vitamin C tablet and 6 pieces of Vitamin E capsule per 100g of gel; F2 (fortified), made up with antioxidant mixture of 8 pieces of Vitamin C tablets and 12 pieces of Vitamin E capsules per 100g of gel; F3, signifies formulation without preservatives and antioxidants; TFTC, too few to count; TNTC, too numeric to count.

Conclusion

This present study was aimed at preparing a low cost facial turmeric gel. So in this work the gel was made with mandatory excipients purchased from corner shops. Such approach avoided buying of ingredients in a bulk and also it did not involve buying an ingredient according to a company's supplying amount (i.e. packaging unit) which seemed 'must'. This surely saved money and met our aim which was to formulate and prepare a low cost product. Secondly considering the fact a new gate way for formulating and preparing stable gels usable for 14–21 days with vitamin C and vitamin E capsules instead of using more costly preservatives like parabens is possible to open up even when the formulations are kept under normal condition.

Conflicts of interests

None.

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