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Formulation and Evaluation of Halal Hair Growth Promoting Shampoo Containing *Centella asiatica* and *Phyllanthus emblica*

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Abstract

Halal awareness among Muslim consumers has widened to a wide range of products including personal care and cosmetics. Shampoo is the most frequently prescribed treatment for hair and scalp conditionswhile *Centella asiatica* and *Phyllanthus emblica* have been found to have antioxidant properties thatexhibit positive benefits on hair. The aim of this study is to develop halal hair growth promoting shampoo containing *P. emblica* and *C. asiatica*. Halal certificate, Certificate of Analysis and Material Safety Data Sheet were requested from the suppliers for halal evaluation of the ingredients. Seven shampoo formulations (F1-F7) were created with varying concentrations of *P. emblica* and *C. asiatica* extracts, while one formulation was left as a blank. Then, the shampoo formulations underwent an organoleptic evaluation along with other tests for dirt dispersion, pH, solid content, foaming capacity, and stability. The 2,2-diphenyl-1-picrylhydrazyl (DPPH) method was used to measure the antioxidant activity. All of the ingredients used to formulate the shampoo are considered halal based on certificate, origin, and their composition. The evaluation of the shampoo demonstrated that all the formulations exhibited ideal physicochemical properties for hair cleansing and F3 with 4% of *C. asiatica* extracts and 2% of *P. emblica* extract produced the best antioxidant activity as it inhibited 92.5% of DPPH. Overall, this study provides the requirement on formulation of halal hair growth promoting shampoo containing *P. emblica* and *C. asiatica*. The results show that the formulated shampoo is halal based on document review, exhibited high antioxidant properties for hair growth promoting effect and possess good characteristics as cleansing agent.

Keywords: Halal, shampoo, antioxidants, hair growth, *Centella asiatica, Phyllanthus emblic*

Introduction

Halal is an Arabic word that refers toany object or an action which is permissible to use or engage in, according to Islamic law, whereas haram is anything unlawful or forbidden(1). Muslim consumer halal awareness has widened from being concerned with meat-based products to a wide range of products today where recent research has cited that more than 20% of Muslim consumers are concerned about halal issues with the products they are using.

Cosmetic products refer to any substance or preparation intended for application to any external part of the human body or the teeth or buccal mucosa mainly for the purpose of cleaning, promoting attractiveness, perfuming, or protecting and keeping them in good condition (2).The halal aspects of cosmetics cover ingredients, safety and all the processes involved in production right up to delivery to consumers.

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A basic concept shared by most halal certifiers of personal care products is vigilance regarding ingredients and their origins, as well as the manufacturing process, to check for potential points of contact with haram products (3).

Shampoo represents the largest segment of hair cosmetics (4) and the treatments are the most commonly used means of managing hair and scalp conditions(5). Shampoo helps to remove dirt, oils, dandruff, skin particles, and other contaminants that gradually build up in hair as well as leaving the hair in a satisfactory condition after rinsing so that it can be combed easily both in the wet and dry state (4).

Hair is derived from the ectoderm of the skin and hair loss affects millions worldwide due to aging, hormonal dysfunction, medications, and supplements, or as a side effect of cancer treatment (6). Molecules that can promote hair follicle stem cell activation have been intensely searched for, as they may help provide therapeutic and cosmetic interventions(6). The drugs of synthetic origin that are approved by the FDA for hair growth are associated with potential side effects (7). Thus, people are interested in the usage of alternative remedies which are herbal hair growth formulations(8).

Medicinal herbs producing good antioxidant activities have been employed as the source of natural antioxidants (9). Studies show that there is a strong connection between antioxidant activity and hair growth promoting effects. The best ingredients are antioxidants that can interrupt radical chain processes, fight free radicals in our body that cause the hair follicle cells in the scalp to break down, help to repair hair systems, protect against oxidative damage as well as increase the blood circulation and thus help in hair growth(10).

Centella asiatica is a medicinal plant that has been used for hundreds of years due to its health-promoting effects, especially in dermatological conditions. It can prevent hair loss as polyphenol, flavonoid, and vitamin C compounds are abundant in *C. asiatica*, contributing to its significantly higher antioxidant activity (11).

Phyllanthus emblica highly contains vitamin C and low molecular weight hydrolysable tannins making it a good source of antioxidant (12). Other than helps to maintain vernal hair color and retards premature graying, it also supports the strength of the hair follicles, so there is less thinning of hair with age(13).Although there are many treatments available, many people are still suffering from hair loss. Therefore, it is important to develop novel shampoo formulations that prevent hair loss and promote hair growth (8). At the same time, Muslims would want to be certain that the cosmetic and personal care products they use are halal (1). *P.emblica*has shown prominent antioxidant effects which can promote hair growth and prevent graying of the hair. *C. asiatica* contains essential oils, sterols, flavonoids, glycosides, and triterpenoid saponins and is commonly used in hair care formulations. It is high in antioxidants hence helps to prevent greying of hairs as well as promote hair growth. However, the combination of *P. emblica*and *C. asiatica* in shampoo formulation is not yet available in the market.

Materials and Methods

Materials

C. asiatica extract was bought from Xi'an SR Bio Engineering in China, while *P. emblica* fruit powder was bought from Bio Organic in Sri Petaling, Kuala Lumpur.Sodium lauryl ether sulphate, polysorbate-20, DMDM hydantoin and EDTA were obtained from Ken Prima (Malaysia). Cocamidopropyl betaine and sodium chloride were purchased from Personal Formula Resources (Malaysia). 2,2-Diphenyl-1-picrylhydrazyl (DPPH), ethanol and ascorbic acid were purchased from R&M Chemicals (Malaysia).

Methods

Identification of halal ingredients

Three documents that were requested together with each ingredient are Halal Certificate, Certificate of Analysis and

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Material Safety Data Sheet. Hence, the ingredients that are halal certified by JAKIM or recognized by JAKIM were preferred.

Preparation of shampoo

Preparation of *P. emblica* extract

The extract of *P.emblica* was prepared using a decoction method. As shown in (Table 1), specific amounts of P. emblica powder and water were used for each shampoo formulation. Following a procedure adapted from previous research (14) with minor modifications, the powder was first dissolved in water in a beaker. The mixture was then heated to 65 °C for 15 minutes and allowed to cool. After cooling, the herbs were filtered out to obtain the extract. This extract was then concentrated to one-fourth of its original volume. The final prepared extract was subsequently added to the base shampoo formulation.

Table 1: Preparation of *P. emblica* extract

Formulation	*P. emblica* powder (g)	Distilled water (ml)
F1	-	-
F2	1	10
F3	2	20
F4	3	30
F5	4	40
F6	5	50
F7	6	60

Preparation of blank shampoo and incorporation of plant extracts

The formulation of the blank shampoo was adapted from previous research (15) with some modifications. Seven shampoo formulations (F1-F7) were created with varying concentrations of *P. emblica* and *C. asiatica* extracts, while one formulation was left as a blank. Initially, SLES, cocamidopropyl betaine, and polysorbate-20 were combined and dissolved in water with continuous stirring. Next, DMDM hydantoin and EDTA were added and stirred until a uniform solution was achieved. The powdered extracts were then dissolved in water and incorporated into the formulation (Table 2). The pH of the mixture was

Table 2: The composition of ingredients used in the formulation

Ingredients	Function	Blank	F1	F2	F3	F4	F5	F6	F7
Distilled water	Solvent	q.s. 100	q.s. 100	q.s. 100	q.s. 100	q.s. 100	q.s. 100	q.s. 100	q.s. 100
SLES	Primary surfactant	20	20	20	20	20	20	20	20
Cocamidopropyl betaine	Secondary surfactant	10	10	10	10	10	10	10	10
Polysorbate-20	Solubilizer	6	6	6	6	6	6	6	6
DMDM hydantoin	Preservative	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
EDTA	Chelating agent	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
Sodium chloride	Thickener	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8
C. asiatica	Active ingredient	-	6	5	4	3	2	1	0
P. emblica		-	0	1	2	3	4	5	6

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adjusted using TEA or citric acid, and finally, sodium chloride was added to adjust the viscosity.

Evaluation based on physicochemical properties

Organoleptic properties

The formulations will be evaluated in terms of their clarity, color, odor, homogeneity, viscosity, and consistency(16).

pH test

The pH meter is calibrated using standard buffer solution, pH of 10% v/v shampoo solution in distilled water was measured at room temperature. The pH of tested commercial shampoos was found within the preferred range between 5.5 to 6.5.(1).

Foaming ability and stability

Foaming ability was determined by using the cylinder shake method. Briefly, 50 mL of the 1% of formulated shampoo solution was placed into a 250 mL graduated cylinder; it was covered with one hand and shaken 10 times. The total volume of the foam content after 1 min of shaking was recorded. Foam stability was evaluated by recording the foam volume after 1 minute and 4 minutes of shake test(17).

Dirt dispersion

Two drops of shampoo were added in a large test tube containing 10 ml of distilled water. I drop of India ink was added, the test tube was stoppered and shake it ten times. The amount of ink in the foam was estimated as None, Light, Moderate, or Heavy (18, 19).

Solid content

A dry porcelain dish was taken, and 4 grams of each shampoo formulation is poured in it. The exact weight of the porcelain dish was noted. The dish is then put for evaporation in the oven at a temperature of 105°C for 3 hours till the whole liquid has evaporated. The amount of solid left drying was determined (18, 20).

Antioxidant study

Antioxidant activity of the shampoo formulation is examined on the basis of scavenging effect and hydrogen donating ability on the stable of 2,2-diphenyl-1-picrylhydrazyl (DPPH) free radical. Ethanol is used as blank sample and ascorbic acid as positive control. The antioxidant activity is determined by using the equation to determine the reduction in absorbance at various concentrations. The method of sample and ascorbic acid preparation are adapted from Joshi *et al*. (2018)(17).

Preparation of sample

A 10 mg/ml shampoo formulation was prepared using ethanol as a solvent, with the volume adjusted to 10 ml in a volumetric flask. A 0.004% DPPH solution was made by dissolving 0.4 mg of DPPH in 100 ml of 70% ethanol. To each sample, 1.5 ml of the prepared DPPH solution was added to 0.5 ml of the shampoo formulation and vortexed. The mixed solution was then placed in a dark room at room temperature for 20 minutes. After this period, UV spectroscopy at 520 nm was used to measure the absorbance of each solution. The free radical scavenging activity of DPPH was calculated using the formula:

$$\% \text{ inhibition} = \frac{\text{A DPPH} - \text{A Sample}}{\text{A DPPH}} \times 100$$

Where,
A DPPH = Absorbance of DPPH
A Sample = Absorbance of sample
Each measurement was repeated three times.

Preparation of Ascorbic acid

A 10 mg/ml solution of ascorbic acid was prepared in ethanol. From this solution, 5 ml were taken and diluted to a final volume of 50 ml. Various concentrations were then prepared from this stock solution, specifically 20 µg/ml, 40 µg/ml, 60 µg/ml, 80 µg/ml, and 100 µg/ml.

Results and Discussion

Evaluation of Halal Status of Shampoo Ingredients

Three documents that were requested upon purchasing the ingredients

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are Halal certificate, Certificate of Analysis (CoA), and Material Safety Data Sheet (MSDS). The list of documents that are available and provided by the suppliers is shown in (Table 3).

The organizations recognized by JAKIM are listed in The Recognized Foreign Halal Certification Bodies and Authorities as of December 1st, 2020. SLES, polysorbate 20, DMDM hydantoin, EDTA, and sodium chloride are considered halal ingredients as they are halal-certified by the organizations that are recognized by JAKIM. According to the documents, the composition of this ingredient is 35% CAPB and 65% water and does not contain animal-derived products. Thus, this ingredient is considered halal. *C. asiatica* extract is halal certified by IFRC Hong Kong. This organization strictly follows the Malaysian Standard and World Halal Council Standard as well as received recognition by Islamic Religious of Singapore (MUIS) and Korea Ministry of Food & Drug Safety (KMFDS). The *P. emblica* extract is handpicked, 100% certified organic by ECOCERT, USDA, India Organic, and FSSAI, purely natural and safe for consumption. According to the website, this extract is also cruelty-free and vegan. It has not been tested on animals and contains no animal parts or extracts.

This study lack of information regarding the manufacturing process of each ingredient. Knowing the origin of raw materials and the production process of cosmetic ingredients is vital for Muslim consumers(21). As a result, some excipients were purchased despite the fact that they did not have a halal certificate and no information on the manufacturing process. Thus, CoA and MSDS were required to ensure that the ingredients' origin, safety, and composition meets the product quality standards.

Evaluation of shampoo

Organoleptic properties

The formulated shampoos were evaluated for physical characteristics such as color, odor, homogeneity, viscosity, and consistency (Table 4). Figure 1 shows organoleptic properties of the shampoo formulations.

Based on Figure 1, the addition of extracts turned the clear shampoo into brown due to the colour of the extracts. F1 (6% of

Table 3: List of documents received from the supplier for halal evaluation

Ingredients	Halal Certificate		CoA	MSDS
	Availability	Organization		
SLES	/	The Central Islamic Council of Thailand*	X	X
Polysorbate-20	/	Islamic Religious Council of Singapore*	X	X
Cocamidopropyl betaine	X	-	/	/
DMDM hydantoin	/	The Indonesian Council of Ulama*	X	X
EDTA	/	Halal Quality Control*	X	X
Sodium chloride	/	ARA Halal Certification Services Centre Inc.*	/	/
C. asiatica	/	Islamic Food Research Centre, Hong Kong	/	/
P. emblica	X	-	X	X

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Table 4: Organoleptic properties of formulated shampoo					
Formulation	Colour	Odour	Homogeneity	Viscosity	Consistency
Blank	Clear	Good	Good	Good	Liquid
F1	Dark brown	Good	Good	Good	Liquid
F2	Dark brown	Good	Good	Good	Liquid
F3	Dark brown	Good	Good	Good	Liquid
F4	Dark brown	Good	Good	Good	Liquid
F5	Dark brown	Good	Good	Good	Liquid
F6	Dark brown	Good	Good	Good	Liquid
F7	Light brown	Good	Good	Good	Liquid

Figure 1: Physical appearance of formulated shampoo

CAE) has the darkest colour while F7 (6% of PEE) has the brightest colour among all formulations. However, there were no trend in changes of colour between F2 to F6 which might be due to a little difference in extract content across the shampoo formulations. No significant difference was observed in terms of odor, homogeneity, viscosity, and consistency between all the formulated shampoo except for color and all the formulations show good characteristics. These parameters were evaluated by vision and touch sensation.

pH

The pH level of all formulated shampoo containing different concentrations of *C. asiatica* and *P. emblica* are tabulated in (Table 5). The pH range of hair is 4.5-5.5 and the optimum pH for shampoo is 5.5 to 6.5(1).

Based on the results, pH levels of all formulated shampoo containing different combinations of *P. emblica*and *C. asiatica* extract are acidic compared to the blank shampoo which is more alkaline. The pH level of F1, F2, and F7 are within the desired range, while pH of F3, F4, F5, and F6 are slightly acidic and below the optimum range. The pH level of all the tested shampoos is slightly acidic compared to the blank shampoo formulation due to the acidic nature of the extracts. F1 and F7 are single formulation of shampoo containing *C. asiatica* and *P. emblica* extracts respectively. It shows that combination of the extracts produced more acidic shampoo instead of its single formulations. One method for reducing hair damage is to adjust the pH. The pH of the acidic shampoo can be adjusted to the optimum level by the addition of triethanolamine solution to prevent any irritation and damage due to the consumption of the shampoo that is too acidic (22).

Foaming ability and stability

Results of foaming ability and stability test for all formulated shampoos are shown in (Figure 2). From the results, generally, the volume of foam produced by F1, F4, F5, F6, and F7 remain constant, while F2 and F3 only show slight changes after four minutes of observation. F3 (4% of CAE 2% of PEE) has the highest foam volume but it slightly decreases after four minutes. F5 (2% of CAE 4% of PEE) has the lowest foam volume but it has good stability as the volume remains constant over the four minutes of observation. On the other side,

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Table 5: Result on pH test of formulated shampoos (n=3)			
Formulation	Concentration of extract (%)		pH (Mean ± SD)
	P. emblica	*C. asiatica*	
Blank	0	0	8.46± 0.02
F1	0	6	5.93 ± 0.01
F2	1	5	5.54 ± 0.01
F3	2	4	5.10 ± 0.00
F4	3	3	4.78 ± 0.00
F5	4	2	5.13 ± 0.00
F6	5	1	4.83 ± 0.01
F7	6	0	5.75 ± 0.01

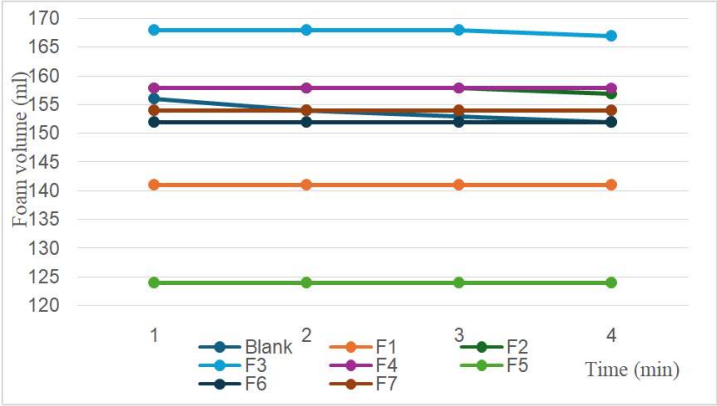

Figure 2: Foam retention profiles of tested shampoos

the blank shampoo formulation also performed good foaming ability, but the foam volume was slowly reducing after two minutes. The foam generated by the prepared shampoos were uniform, compact, and stable.

Based on the graph, it shows that all formulations have good foaming ability and stability. However, in comparison to the blank shampoo, all the shampoo formulations containing the extracts have better foaming stability even though the difference was not significant. In addition, F4 (3% of CAE 3% of PEE) has the second highest foam produced and the foam remains constant over the four minutes of observation. Thus, it indicates that the addition of both extracts into the shampoo formulation does not adversely affect its foaming ability and increase its stability instead. The higher foaming property of formulated shampoos may be due to the sufficient amount of surfactants(23).

Solid content

Table 6 shows the mean value of solid content evaluation conducted on the prepared shampoos. The best range is between 20-30%. The total solid content of shampoo indicates its ability to clean. According to Badi & Khan (16), the best range is between 20-30% because it is easy to apply and rinse out of hair. A shampoo is considered good if the percentages of solid contents are good enough to cause easy

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Table 6: Result on total solid contents of all formulated shampoos (n=3)			
Formulation	Concentration of extract (6)		Solid content (Mean ± SD)
	P. emblica	*C. asiatica*	
Blank	0	0	16.66 ± 1.76
F1	0	6	17.50 ± 2.46
F2	1	5	19.08 ± 3.13
F3	2	4	13.50 ± 0.66
F4	3	3	15.17 ± 1.26
F5	4	2	14.33 ± 1.26
F6	5	1	11.25 ± 0.90
F7	6	0	11.75 ± 1.00

Figure 3: Result of dirt dispersion test

application and removal from the hair. The percentage of total solid contents for all formulated shampoo was found within the range of 11-19% which is less than 20%, the required range. The amount of solid in all of the tested shampoos was deemed insufficient, and the shampoos were expected to be washed out of the hair very quickly(20). However, there is no survey that demonstrates customer satisfaction with adequate number of solid contents in a shampoo. According to observations, the prepared shampoos have a suitable viscosity and are not too watery. All of the excipients used to make the shampoo are water soluble, which could explain why the solid content is insufficient.

Dirt dispersion

One of evaluations that can be done to assess the cleansing action of shampoo is dirt dispersion. The ink should remain in the water portion to be considered as good quality and has better cleansing action. Figure 3 shows the result for dirt dispersion test for all formulated shampoos.All shampoo concentrated the ink in the water portion.

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Table 7: Results on DPPH scavenging activity of different shampoo formulations (n=3)

Formulation	Concentration of extract		Percentage of scavenging activity (Mean ± SD)
	P. emblica	*C. asiatica*	
F1	0	6	27.37 ± 7.12
F2	1	5	58.82 ± 3.20
F3	2	4	92.52 ± 1.43
F4	3	3	84.65 ± 1.12
F5	4	2	88.80 ± 2.01
F6	5	1	77.17 ± 1.88
F7	6	0	90.46 ± 5.13

Shampoos that cause ink to concentrate in the foam are considered low-quality because ink or dirt that remains in the foam is difficult to rinse away and re-deposits on the hair (16). All shampoo concentrated the ink in the water portion, ensuring adequate cleaning potential and actual effectiveness. This might be due to the samenumber of surfactants which act as a cleaning agent in all the seven formulations. Therefore, the prepared formulations are satisfactory.

Antioxidant study

Shampoo formulations were prepared containing combination of *C. asiatica* and *P. emblica* extract of different concentrations and their antioxidant activity were determined by using DPPH method. UV spectroscopy was used to measure the percentage of scavenging effect produced by the formulated shampoos with absorbance measured at 520 nm (17). Table 7 shows the mean percentage of scavenging activity of the tested shampoos.

Based on the result, F1 (6% of CAE) inhibits 27.4% of DPPH which indicates that the single shampoo formulation containing *C. asiatica* extract demonstrated low antioxidant properties. F7 (6% of PEE) inhibits 90.5% of DPPH which indicates that the single shampoo formulation containing *P. emblica* extract shows a high antioxidant activity compared to other formulations. However, the percentage of inhibition of the shampoo combination F3 (4% of CEA and 2% PEE) is 92.5% which is the highest among all tested shampoos. In comparison to ascorbic acid (100µg/ml), which inhibits 99.28% DPPH, this result demonstrates that the combination of both extracts in the ratio of 2% of *P. emblica* and 4% of *C. asiatica* extract in shampoo produces high antioxidant activity. Compared to F1 (6% of CAE), other combination formulation of shampoo also demonstrated high DPPH scavenging activity. Thus, it shows that the shampoo formulation containing combination of both extracts are better than CAE alone and F3 are the highest among all formulated shampoo.

In shampoo containing combination of extracts, different ratios of the extracts produced different antioxidant properties. This is supported by previous research conducted by Joshi *et al*. (17) where the ratio of betel leaves: guava leaves extract of 1:3 g possessed the highest antioxidant properties. This present study shows that F3 inhibited the highest percentage of DPPH compared to other combination formulations, F2, F4, F5 and F6 and comparable to ascorbic acid standard.However, there are no studies that investigate the effect of the combination of these extracts on antioxidant activity and limited antioxidant studies conducted on shampoo formulations. Previous research concentrated solely on plant extracts. Thus, this study has shown that the combination of *P. emblica* and *C. asiatica* extracts in a shampoo produce high antioxidant effects compared to their single formulations.

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Conclusion

The present study aimed to formulate halal hair growth promoting shampoo containing combination of *C. asiatica* and *P. emblica*. All the ingredients used in this study are considered halal based on the origin of each ingredient. Physical appearance, pH, foaming ability and stability, solid content, and dirt dispersion of the shampoo combination were evaluated and compared to the blank shampoo and their single formulations. The results show that the shampoo combinations were not significantly different to the blank and single formulations, instead it has better foam stability. Based on the antioxidant study using DPPH method, F3 (4% of CAE 2% of PEE) shows the highest percentage of scavenging activity compared to other formulations. As a result, the findings of this study have provided the basic information on the feasibility of formulating a halal hair growth promoting shampoo containing combination of *C. asiatica* and *P. emblica* extracts. The blank shampoo formulation produced ideal physicochemical properties and it was not adversely affected by the addition of both extracts. The combination of shampoo (F3) demonstrated high antioxidant properties comparable to the ascorbic acid standard. These findings are important as the combination formulation is not currently available in the market yet and has the potential to be marketed in the future.

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