

Powder shampoo formulation with antioxidant activity: in vitro perspectives

Powder
shampoo
formulation

Aishwariya Madhavan, Meher Unnati, K. Rachana, Prateek Jain,
K. Bhashasaraswathi and Apurva Kumar Joshi
*Department of Chemistry and Biochemistry, School of Sciences,
Jain (Deemed to be University), Bangalore, India*

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Abstract

Purpose – The purpose of the study was to develop a powder shampoo with antioxidant attributes.

Design/methodology/approach – Dry shampoo compositions were formulated containing alpha olefin sulfonate (AOS), sodium cocoyl isethionate (SCI), microcrystalline cellulose, mannitol, carboxymethyl cellulose, maltodextrin and sodium benzoate with or without extract of *Cinnamomum zeylanicum* bark. Cinnamon extract was chosen for this study owing to its ubiquitously known antioxidant attributes. The formulations were tested for detergency action and antioxidant potential in vitro.

Findings – *Cinnamomum zeylanicum* extract exhibited noticeable antioxidant activity in vitro. The authors observed that addition of the bark extract to the shampoo formulation was associated with remarkable increase in total phenolic content, total antioxidant activity and radical scavenging activity without any effect on detergency action.

Research limitations/implications – This preliminary study provides a powder shampoo formulation which exhibits antioxidant attributes as a result of incorporation of cinnamon bark extract. Clinical efficacy of the formulation remains to be tested.

Practical implications – Owing to the powder format of the shampoo, the formulation can be manufactured with ease and economically. Functionalizing the formulation with enhancement of antioxidant activity by incorporation of cinnamon bark extract may be associated with beneficial clinical outcomes, which remains to be tested.

Social implications – The proposed formulation may be stored and sold in eco-friendly packing material, thus could pave the way for reducing the burden of plastic consumption by the shampoo industry.

Originality/value – The present work demonstrates that incorporation of cinnamon bark extract to a powder shampoo formulation, containing AOS and SCI as principle surfactants, significantly enhances its antioxidant attributes.

Keywords Alpha olefin sulfonate, DPPH radical scavenging, *Cinnamomum zeylanicum*, Powder shampoo, Sodium cocoyl isethionate, Total phenolic content

Paper type Technical paper

Introduction

Hair acts as a defense barrier against external factors (Erdoğan, 2017). This is particularly true in the case of scalp hair offering protection against UV radiation (Zaidi & Lanigan, 2010). Despite vital role of scalp hair as defensive barrier and its contribution to overall physical appearance, it is believed that hair care is not taken as seriously as skin care (Trüeb, 2021). Hair shaft majorly consist of cortex, cuticle cells and medullary cells, whereas hair follicle is essential for the



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generation of hair. The hair follicle follows a pattern of continuous growth and rest cycle (Erdogan, 2017), which are shown to be influenced by various internal and external factors such as endocrine, neural stimuli, vascular, age, nutritional habits and maintenance of personal hygiene (Trüeb, 2021). The effects of pollutants, dust, chemicals and ultraviolet radiation exposure onto the scalp leads to hair damage via the generation of reactive oxygen species. Reactive oxygen species are a term used to describe highly reactive intermediates produced in cells by various mechanisms (Hoting & Zimmermann, 1996; Nogueira, Dicelio, & Joekes, 2006; Signori, 2004). These intermediates, when not sufficiently countered by antioxidant defenses, could cause oxidative damage to cellular components and such damages have been implicated in the etiology of many disorders affecting key organs such as cardiovascular system, kidney and nervous system (Bandyopadhyay, Das, & Banerjee, 1999; Liguori *et al.*, 2018). Therefore, it is not surprising that oxidative stress has been studied as possible mediators of dyshomeostasis associated with hair health. Upton *et al.* showed that dermal papilla cells (DPC) from balding men are more sensitive to oxygen and secrete higher levels of negative hair growth regulators in response to hydrogen peroxide than occipital DPCs. Further, DPCs from balding men were reported to be less adept at handling oxidative stress in spite of higher levels of catalase and glutathione levels (Upton *et al.*, 2015). Androgenetic alopecia patients have been reported to be associated with lower levels of superoxide dismutase and higher levels of lipid peroxidation in plasma along with reduced total antioxidant capacity (TAC) (Prie, Iosif, Tivig, Stoian, & Giurcaneanu, 2016). 8-hydroxy deoxyguanosine (8-OHdG), a marker of oxidative DNA damage, and C-reactive peptide are reported to be higher in serum of patients afflicted with alopecia areata (Mustafa, Khashaba, Fawzy, Baghdady, & Rezk, 2021). Acharya and Mathur in their review provide an account of investigations that suggest that alopecia areata is associated with oxidative stress, and magnitude of oxidative stress correlates with severity of alopecia areata (Acharya & Mathur, 2020). Peterle *et al.* suggest that malondialdehyde and advanced glycated end products could serve as biomarkers for alopecia areata (Acharya & Mathur, 2020).

Shampoos are cosmetic preparations used in cleansing hair and scalp as most of the contaminants are water insoluble thereby requiring efficient surfactants to remove those (Robbins, 1998). Water-based shampoos have been very prevalent in the market space since a long time. One of the primary and vital concerns over the use of liquid shampoo is the utilization of plastic bottles and sachets for the storage. Macroplastics have been well recognized as contaminants in aquatic domain and are known to cause tremendous physical injuries and even death due to either entanglement or ingestion (Cole, Lindeque, Halsband, & Galloway, 2011; Derraik, 2002; Ryan, 1987, 2008). There are concerns over breakdown of macroplastics into microplastics and effects of the latter on ecosystem. It suggest that they can act as a possible threat to the ecosystem as these small plastic fragments were found in the digestive system of birds, crustaceans, turtles, seabirds and fish (Cole *et al.*, 2011; Derraik, 2002; Ryan, 1987, 2008). Accumulation of such small fragments plastic in the gut further leads to starvation, malnourishment and premature death of the affected species (Boerger, Lattin, Moore, & Moore, 2010; Ryan, 2008). Therefore, it is highly desirable to use biodegradable materials in packing and distribution of personal care products. Furthermore, certain chemicals used in market available shampoo such as zinc pyrithione, selenium sulfide are known to cause damage to the keratinocytes (Gilbertson, Jarrett, Bayliss, & Berk, 2012; Lamore, Cabello, & Wondrak, 2010). These could also cause damage at molecular level thereby could be potentially genotoxic in nature (Lamore *et al.*, 2010). Because of the abovementioned reasons, herbal shampoo is becoming widely acceptable due to the incorporation of plant-derived substances (Godeto *et al.*, 2023; Saripalla *et al.*, 2021). The secondary metabolites of plant extracts are known to be enriched with vitamin C, vitamin E, vitamin B, polyphenols, flavonoids, tannins, carotenoids and organosulfur compounds (Godeto *et al.*, 2023; Sharifi-Rad *et al.*, 2020), which serve to counter the effects of pro-oxidants, thereby preventing pathologies associated with oxidative stress (Godeto *et al.*, 2023; Saripalla *et al.*, 2021; Sharifi-Rad *et al.*, 2020) as well as aiding in improving

hair strength, hair texture, volume and hair growth. Usage of herbs such as aloe vera (Chaitanya & Jaiswal, 2022), Acacia concina (shikakai) (Saripalla *et al.*, 2021), Azadiracta indica (Neem) (Chaitanya & Jaiswal, 2022), Cyclea peltata (raj patha) (Cheni Cheri, Kizhakke Veetil, Pradeep, & Nayak, 2022), Ocimum temple (tulsi) and Phyllanthus emblica (amla) (Saripalla *et al.*, 2021) has proven to be highly beneficial not only as surfactant but also resolving several scalp concerns such as dandruff, seborrhea dermatitis in the shampoo formulation (Chaitanya & Jaiswal, 2022; Cheni Cheri *et al.*, 2022; Saripalla *et al.*, 2021). It could be argued that hair cleansing formulation containing surfactants and functional ingredients in dry or powder format offer the advantage of reduced cost of manufacturing and storage in environmentally friendly containers.

Given the need for powder shampoos and fictionalization of shampoos with antioxidant attributes, the present study developed a powder shampoo formulation containing *Cinnamomum zeylanicum* bark extract, alpha olefin sulfonate (AOS) and sodium cocoyl isethionate (SCI) for evaluation of detergency action and antioxidant potential *in vitro*.

Materials and method

Chemicals

Ammonium molybdate, ascorbic acid, disodium hydrogen phosphate, FC reagent, gallic acid, hexane, methanol and sodium bicarbonate were obtained from Sisco Research Laboratories (India). AOS was procured from BRM Chemicals, Rajasthan, India. Mannitol and maltodextrin were procured from Bioven Ingredients Pvt. Ltd., UP, India. Microcrystalline cellulose was from Accent Microcell Pvt. Ltd., Gujrat, India. SCI was from Pratha, India. Cinnamon bark was procured from local market. All other chemicals used in the present study were of analytical grade.

Extraction

Cinnamomum zeylanicum bark powder was extracted twice with methanol at room temperature. Supernatants obtained by filtration were pooled and concentrated under vacuum at 45°C to obtain a pleasant-smelling dark-colored viscous oleoresin (Mathew & Abraham, 2006).

Powder shampoo formulations

Powder shampoo containing extract of bark of *Cinnamomum zeylanicum* (DSCE) was prepared by mixing AOS, SCI, microcrystalline cellulose mannitol, carboxymethyl cellulose, maltodextrin, methanolic extract of bark of *Cinnamomum zeylanicum* and sodium benzoate. The details regarding the composition are given in Table 1. Above described ingredients were

Sl No	Ingredient	Purpose	Amount (g)
1	Alpha olefin sulfonate	Surfactant	40.0
2	Sodium cocoyl isethionate	Surfactant	20.0
3	Microcrystalline cellulose	Anti-Cracking agent	10.0
4	Mannitol	Anti-Cracking agent	10.0
5	Carboxymethyl cellulose	Rheology modifier	10.0
6	Maltodextrin	Filler	8.7
7	Methanolic extract of bark of <i>Cinnamomum zeylanicum</i>	Source of antioxidant activity	1.2
8	Sodium benzoate	Preservative	0.1

Source(s): Table by authors

Table 1.
Dry shampoo
formulation

mechanically blended to obtain a homogenous powder. For comparison of antioxidant potential, a formulation designated as “DS” was prepared by replacing the methanolic extract of bark of *Cinnamomum zeylanicum* with maltodextrin.

Assays

For evaluation of antioxidant potential *in vitro*, dry shampoo containing extract of bark of *Cinnamomum zeylanicum* (DSCE) and dry shampoo without extract of bark of *Cinnamomum zeylanicum* (DS) were extracted with methanol, and the methanol soluble fraction was tested for radical scavenging activity, total phenolic content and TAC as described below. The cinnamon extracted was dissolved in methanol for evaluation of antioxidant potential *in vitro*.

Total phenolic content

Folin-Ciocalteu method was used for the estimation of the phenolic content in of the dry shampoo formulations. The phenolics in the extract reduce the reagent to produce molybdenum–tungsten blue, which is read at 765 nm, intensity of the color produced was found to be in linear relationship with the concentration of phenolics. The experiment was carried out in 96 well plate. The assay was initiated by the addition of 85µl of distilled water followed by 10µl of sample (methanolic extracts) and 25µl of FC reagent (1:1 dilution). Post an incubation period of 6 minutes, 100µl Na₂CO₃ (75g/l) was added and plate was in dark for 90 min. The plates were read at 765 nm using a microplate reader. Gallic acid was used as standard and results were expressed as gallic acid equivalents (Herald, Gadgil, & Tilley, 2012).

Total antioxidant activity (TAC)

The TAC of the test extract was spectroscopically determined using phosphomolybdenum method, which is based on the reduction of Mo (VI) to Mo (V) complex under acidic conditions. 50µl of the samples was mixed with 500µl of the reagent (28 mM sodium phosphate, 4 mM ammonium molybdate and 0.6M sulfuric acid) and kept in boiling water bath for 90 mins. After cooling to room temperature, the absorbance was measured at 695 nm. Ascorbic acid was used to obtain the calibration curve (Alam, Bristi, & Rafiquzzaman, 2013).

Radical scavenging activity

The radical scavenging activity exhibited by dry shampoo formulations was determined by monitoring scavenging of the free radical α , α -diphenyl- β -picrylhydrazyl (DPPH) spectrophotometrically. 100µl of DPPH reagent (8mg in 100 ml methanol) was incubated along with 100 µl of sample (ascorbic acid or dry shampoo formulations or the bark extract) in 96 well plate for 30 min in dark and read at 514 nm (Lee, Oh, Cho, & Ma, 2015).

The detergency action. The detergency action of dry shampoo formulations was evaluated by the Barnet and Powers test with modifications (Patidar, 2018). Briefly 1 g of wool yarn was greased with vegetable oil and weighed. The greased wool yarn samples were then placed in 200 ml of water suspension prepared with one gram of dry formulations. The flasks were shaken for four minutes at 35 degree Celsius. The wool yarn samples were then gently washed with water and dried. Weights of the wool yarns were recorded to determine the detergency and cleaning action attributable to emulsification property of the formulation.

Statistical Analysis: Data have been represented as Mean $\pm$ SD ($n = 3$). ANOVA and Tukey post hoc tests were determined to determine the significances in differences of means.

Results

Antioxidant activity of methanolic extract of bark of *Cinnamomum zeylanicum*

Antioxidant activity of the cinnamon extract is depicted in Table 2. As depicted in the table, the extract possessed appreciable amounts of phenolic content and exhibited strong TAC and radical scavenging activity.

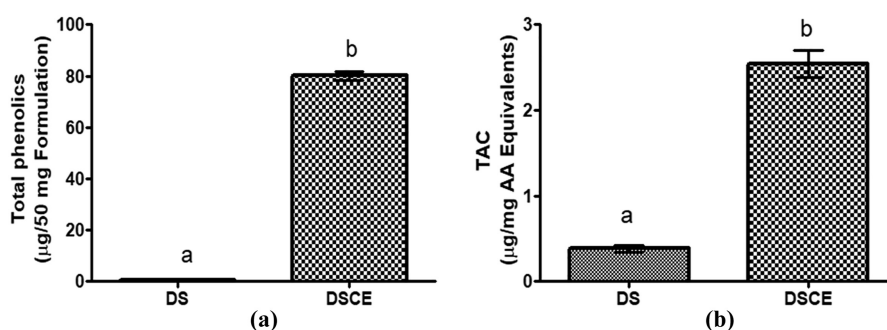
In vitro antioxidant attributes of the shampoo formulations

As depicted in Figure 1A, DS (dry shampoo without cinnamon extract) possessed negligible amounts of phenolics, while DSCE (dry shampoo with cinnamon extract) contained significantly higher amounts of total phenolics. Addition of cinnamon extract to shampoo formulation caused increase in TAC by several folds (Figure 1B). The data on radical scavenging activity of shampoo formulations are Figure 2A. DS (dry shampoo without cinnamon extract) possessed negligible DPPH scavenging activity, while DSCE (dry shampoo with cinnamon extract) was capable of significantly higher DPPH scavenging (>70%). Ascorbic acid was capable of strong DPPH scavenging activity with an EC₅₀ value of 14.0 ± 0.71 $\mu\text{g/mL}$ (data not shown). As depicted in Figure 2B, DSCE and DS were capable of removing more than 95% of oil while water washing (WS) was associated with removal of small amounts of oil (17%). Incorporation of cinnamon bark extract did not affect the detergency efficacy of the dry shampoo formula.

Total phenolic content	Total antioxidant capacity	DPPH scavenging (EC ₅₀)
112.6 $\pm$ 3.4 mg gallic acid equivalents/gram extract	85.44 $\pm$ 0.85 mg ascorbic acid equivalents/gram extract	0.59 $\pm$ 0.02mg/mL

Note(s): Data represented as Mean $\pm$ SD ($n = 3$)
Source(s): Table by authors

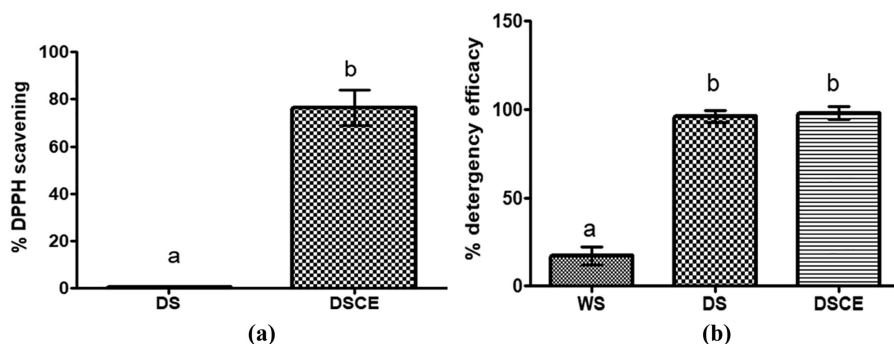
Table 2.
Antioxidant activity of
methanolic extract of
bark of *Cinnamomum*
zeylanicum



Note(s): Data depicted as mean $\pm$ SD ($n = 3$). Bars with different letters are significantly different (t -test, p value < 0.05) (1A). Total antioxidant capacity of dry shampoo formulations. Data depicted as mean $\pm$ SD ($n = 3$). Bars with different letters are significantly different (t -test, p value < 0.05) (1B). DS represents Dry shampoo without cinnamon extract, while DSCE represents Dry shampoo with cinnamon extract

Source(s): Figure by authors

Figure 1.
Total phenolic content
of dry shampoo
formulations



Note(s): Data depicted as mean $\pm$ SD ($n = 3$). Bars with different letters are significantly different (t -test, p value < 0.05) (2A). Detergency action of dry shampoo formulations. Data depicted as mean $\pm$ SD ($n = 3$). Bars with different letters are significantly different (ANOVA and Tukey, p value < 0.05) (2B). DS represents Dry shampoo without cinnamon extract, while DSCE represents Dry shampoo with cinnamon extract and WS represents water wash

Source(s): Figure by authors

Figure 2.
Radical scavenging
activity of dry
shampoo formulations

Discussion

The present study aimed at developing a unique blend of powder shampoo formulation containing cinnamon bark extract as source of antioxidant activity. The strategy of this preliminary work revolved around evaluating the detergency action attributable to a blend of routinely surfactants such as AOS and SCI, and *in vitro* assessment of antioxidant potential attributable to methanolic extract of cinnamon bark. Further, both of these surfactants that is AOS (Blagojevic, 2016) and SCI (Baumann, 2009; Burnett *et al.*, 2017) have already shown to be environmental friendly with no adverse reactions observed on the surface of soil and water (Baumann, 2009; Blagojevic, 2016; Burnett *et al.*, 2017). Both AOS and SCI are commonly used surfactants in cosmetic preparations (Hibbs, 2007). Additionally, SCI is known to be a gentler surfactant action onto the skin and scalp as these aren't capable of penetrating onto the skin layer due to larger micellar structure formations (Ghosh & Blankschtein, 2008; Madhavan & Andersen, 2000). Further, mannitol and microcrystalline cellulose were incorporated as anticaking agents that were also reported to be relatively nonirritant and nontoxic in nature (Rouaz *et al.*, 2021; Weiner & Kotkoskie, 1999). The formulation is designed to be used as a reconstitutable powder that needs to be suspended in water prior to application by the user. Since higher viscosity is a desirable attribute, we also incorporated sodium carboxymethyl cellulose as rheology modifier. Data on *in vitro* detergency action (Figure 2B) the blend of AOS and SCI along with other additives exhibited excellent detergency action. We evaluated whether maltodextrin per se exerts antioxidant effect by assessing its TAC. We observed that the sample of maltodextrin used as filler in the present study possessed the TAC of 1.9 ± 0.16 mg ascorbic acid equivalents/gram (data not shown). Consider that maltodextrin was added at 8.7% in the formulation our data suggest that approximately 0.17 mg ascorbic acid equivalents/gram of the TAC of the formulation containing cinnamon extract is probably attributable to maltodextrin.

Oxidative stress may exert antagonistic effect on hair physiology. Topical treatment of mice with organic peroxide was reported to be associated with early precipitation of the catagen phase of the hair cycle. Further, the study also reported that organic peroxide causes apoptosis in hair follicle cells as well as human epidermal keratinocytes (Hibbs, 2007). Moreover, a clinical trial revealed that application of functional ingredients containing

antioxidants was associated with reduced hair shedding and increased total hair count (Davis *et al.*, 2021). We have provided a brief account of studies reporting on association between oxidative stress and hair and scalp homeostasis in the introduction section of the manuscript. Thus, it may be argued that incorporation of antioxidant attributes may be of benefit for hair care. A patent document reports the application of antioxidant shampoo significantly reduced the levels of oxidative stress marker (hydroxyoctadecenedioic acids) in the scalps (Schwartz *et al.*, 2016). Thus, a shampoo with antioxidant attributes may exert beneficial effects on hair health.

In order to confer the formulation antioxidant capabilities, a methanolic extract of *Cinnamomum zeylanicum* bark was incorporated into the dry powder formulation at 1.2%. *Cinnamomum zeylanicum* has a long history of utility as a traditional medicine (Ranasinghe *et al.*, 2013). Antioxidant attributes of cinnamon have been well documented in studies of various designs (Mancini-Filho, Van-Koij, Mancini, Cozzolino, & Torres, 1998; Mathew & Abraham, 2006; Roussel, Hininger, Benaraba, Ziegenfuss, & Anderson, 2009; Sandamali, Hewawasam, Jayatilaka, & Mudduwa, 2021). *Cinnamomum zeylanicum* is known to contain cinnamaldehyde, α -copaene, α -muurolene, p-methoxycinnamaldehyde and δ cadinene (Abelan *et al.*, 2022). Apart from these, they are also known to be enriched with polyphenols such as catechin, epicatechin, caffeic acids, procyanidin B12, vanillic acid and ferulic acids (Khuwijitjaru *et al.*, 2012). All these primary molecules are known to have antibacterial, antifungal and antioxidant potential (Abelan *et al.*, 2022; Ranasinghe *et al.*, 2013). Recent studies also suggest the use of these extracts to remove dandruff and promote hair growth with improved hair length and density in alopecia patients (Abelan *et al.*, 2022; Mehta, Mehta, & Prasad, 2019). We observed that methanolic extract of cinnamon exhibited appreciable antioxidant activity in vitro (Table 2) and incorporation of the extract into powder shampoo formulation significantly enhanced the antioxidant potential of the powder shampoo (Figures 1A, 1B and 2A). We evaluated total phenolic content of the extract and the formulation containing cinnamon extract as phenolic compounds are known for their antioxidant potential (Miguel-Chávez & Miguel-Chávez, 2017). US10925823B2 describes synergistic antioxidant formulations including that of shampoo (Schwartz, Laughlin, Li, Chen, & Kerr, 2019). US20160346184A1 reveals that levels of hydroxyoctadecenedioic acids are higher in unhealthy scalps and shampoo formulations that decrease hydroxyoctadecenedioic acids in scalp (Schwartz *et al.*, 2016). A kit of shampoo and lotion with anti-inflammatory and antioxidant attributes containing polyphenols from green tea, nanoencapsulated biotechnological blend obtained from Tahiti blue microalgae and Baicapil, flavonoid-rich preparation was reported to increase total follicular unit, number hair and Median hairs per follicular unit (Anzai, Pereira, Malaquias, Guerra, & Mercuri, 2020). However, the above listed formulations do not contain cinnamon extract as source of antioxidant activity. US20220168197A1 describes a powder formulation whose list of ingredients shows *Cinnamomum zeylanicum* as one of many herbal components used. However, patent doesn't describe method of processing *Cinnamomum zeylanicum*. Further, patent doesn't provide data on antioxidant attributes of cinnamon (Battermann & Kerl, 2021). Given traditional use of cinnamon as a spice and its well-known antioxidant attributes, we opine that it is worth exploring the clinical efficacy of the formulation described herein. Several investigators have attempted at developing hair care formulations that are based on antioxidant attributes or aiming at achieving antioxidant outcomes.

Conclusion

Our study reveals a dry powder shampoo formulation with significant antioxidant activity as a result of incorporation of cinnamon bark extract. The antioxidant shampoo formulation with detergents (AOS and SCI) and anticaking agents described in the present study is

appropriate for storage in environmental-friendly packing materials and hence may offer a solution to mitigate burden of use of plastic as storage material for hair cleansing products. The formulation is to be suspended in water just before the use. We opine that the formulation can be explored for antioxidant efficacy in preclinical and clinical settings.

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Corresponding author

Apurva Kumar Joshi can be contacted at: r.apurvakumar@jainuniversity.ac.in

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