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Should Consumers Transfer Inorganic Sunscreens Into Travel-Size Containers? Evaluation of Inorganic Sunscreen Emulsion Performance, Quality, and Stability in a 12-Week Study

AVA M. PERKINS, NONGDO BOUGOUMA AND GABRIELLA BAKI
The University of Toledo, College of Pharmacy and Pharmaceutical Sciences, Department of Pharmacy Practice, Toledo, OH, United States (A.P., N.B., G.B.)

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Synopsis

With COVID-19 restrictions lifting over the past year, consumers feel more inclined to travel, especially to warmer climates. Due to the Transportation Security Administration's regulations, consumers must use smaller-sized personal care products, including sunscreen, by either purchasing travel-sized products or transferring them to alternative packaging that are 3.4 oz or smaller. Doing so may result in stability issues impacting the products' safety and efficacy. The goal of this study was to evaluate the quality, stability, and performance of three commercial inorganic sunscreen emulsions containing zinc oxide, titanium dioxide, and a combination of these UV filters when transferred from their original packaging into four different travel-size (i.e., 2 oz) containers, including clear plastic bottles, clear glass jars, aluminum jars, and silicone bottles. The sunscreens were evaluated for *in vitro* SPF, critical wavelength, spreadability, pH, viscosity, particle size, and physical stability after a 0-, 2-, 4-, 8-, and 12-week period at 25°C and 45°C. Additionally, the samples were subject to three freeze-thaw cycles. The silicone bottles proved to be unsuitable packaging for inorganic sunscreen emulsions due to the loss of material, changes in spreadability, and the substantial increase in *in vitro* SPF values of all three sunscreens. The titanium dioxide-based sunscreens solidified in silicone packaging both at 25°C and 45°C after 2 weeks, and the zinc oxide-based and combination sunscreens' *in vitro* SPF in silicone packaging at 25°C increased by 123% and 636%, respectively, and at 45°C increased by 136% and 766%, respectively. Additionally, the firmness of the zinc oxide-based and combination sunscreen changed significantly in silicone packaging at 25°C during the stability study. While transferring personal care products into smaller, trendy containers may sound like a reasonable option for consumers, doing so could trigger compatibility issues between the packaging and the product. Transferring sunscreens from their original packaging leads to changes in the product's appearance, and most importantly, it can affect the extent of protection they provide.

INTRODUCTION

Since the beginning of the COVID-19 pandemic, the number of traveling passengers recorded by the Travel Security Administration (TSA) has doubled in the past two years.[1]

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*Address all correspondence to Gabriella Baki, Gabriella.Baki@utoledo.edu

Per TSA regulations, liquid cosmetics and personal care items are restricted to only 3.4 oz or less in a passenger's carry-on bag, while liquids over 3.4 oz are only permitted in checked baggage.[2] Since there are weight limitations and extra fees for checked baggage, travelers frequently bring only carry-on luggage and, therefore, must abide by the TSA's carry-on regulations. Purchasing travel-sized (i.e., 3.4 oz or smaller) products after already purchasing full-sized cosmetic and personal care products can be expensive and inconvenient, especially when major retailers such as Walmart and Amazon sell a wide variety of generic travel-sized containers to transfer products in, often marketing them as "TSA-approved."

Sunscreens are over-the-counter drugs in the United States that provide photoprotection against harmful ultraviolet (UV) radiation to prevent sunburn, aging, and skin cancer. Skin cancer, caused by UV radiation, is the most common cancer in the United States, affecting more than 9,000 people per day.[3] Therefore, daily use of sunscreen is essential, and sunscreens are often packaged when traveling.

Inorganic sunscreens have gained popularity over chemical sunscreens in the United States over the past five years due to the combination of legislative changes, safety and environmental concerns, and content created on social media. Under a recent rule proposed by the Food and Drug Administration (FDA) in 2019, only zinc oxide (ZnO) and titanium dioxide (TiO_2) are considered generally recognized as safe and effective (GRASE) in sunscreens.[4] Inorganic sunscreens have also been gaining traction among social media users, with over 151.3 million views under hashtags #mineralsunscreen, #inorganicsunscreen, and #physicalsunscreen on TikTok[5], while hashtags #chemicalsunscreen and #organicsunscreen have 34 million views. Additionally, the term "mineral sunscreen" has been searched up to 376% more on Google than "chemical sunscreen" over the past five years.[6]

It is crucial that sunscreens maintain their integrity over time and keep their consistency, aesthetics, and performance the same. While transferring sunscreens to alternative packaging may seem trendy or convenient when traveling, it may change their overall stability, safety, and efficacy. The aim of this study was to evaluate and compare the stability, quality, and performance of inorganic sunscreen emulsions containing ZnO, TiO_2, and a combination of ZnO and TiO_2 as UV filters in plastic (P), glass (G), metal (M), and silicone (S) packaging during 12 weeks at 2 different temperatures (25°C and 45°C). To our knowledge, this is the first study that focused on the stability of inorganic sunscreens and evaluated four different types of packaging.

MATERIALS AND METHODS

MATERIALS

Three commercial sunscreens currently on the market were purchased from Amazon, including Coppertone® Pure and Simple Kids SPF 50 containing ZnO in 24.08% (Lot #TN00CD0, Beiersdorf, Stamford, CT), Ombrelle® Kids Sunscreen Lotion SPF 50+ containing TiO_2 in 15 % (Lot #20W20Z, Garnier, Montreal, Canada), and Banana Boat® Sport Sunscreen Lotion SPF 50+ containing ZnO in 4.5% and TiO_2 in 6.5% (referred to as combination, Lot #210425117, Edgewell, Shelton, CT). Table SI shows the ingredients in each sunscreen. The intention of selecting sunscreens that were all SPF 50 was to test and compare sunscreens with the same amount of protection. Additionally, caprylyl methicone (Dow, Midland, MI) was used to dilute the sunscreen samples to evaluate particle size.

The packaging materials were also purchased from Amazon, including 2 oz clear plastic bottles with flip caps (ZWLFLF), 2 oz clear glass jars with metal lids (Pinnacle Mercantile), 2 oz aluminum jars with lids (Yvjnxxan), and 2 oz soft silicone squeeze tubes with flip caps (SYBL).

Plastic is the most common packaging for cosmetic and personal care products. Common types of plastic include polyvinyl chloride, typically used for clear plastic bottles; acrylonitrile butadiene styrene, often used for sturdier compact containers; and styrene-acrylonitrile resin (SAN), typically formed into clear jars.[7] The plastic bottle in this study was made of polyethylene terephthalate (PET), and the cap of polypropylene (PP). PET is an aliphatic polyester with great clarity, high flexibility, and good dimensional stability.[8] PP is a type of polyolefin that is physically sturdy and relatively chemically resistant with a low density and high heat resistance.[9] While clear plastic packaging is popular, transparency of the container may allow light degradation of a sunscreen formulation.

Primarily derived from silicate, glass packaging is known to be impermeable to outside contaminates and unlikely to react with the products it contains, making this packaging type an ideal carrier for many cosmetic and personal care products.[10] Additionally, many consumers are turning to products in glass packaging not only because of the more sustainable impact, but also because it enhances the consumer experience with its look and feel.[11] However, its brittleness, fragility, and transparency do not make this material a practical option for manufacturers to produce. While amber or green colors are available to account for light instability, transporting glass packaging is less cost-effective due to its heavier weight and special handling.

Aluminum packaging is lightweight, cost-effective, flexible, and the most common metal-based packaging material for cosmetics. While these properties may be advantageous, other factors need to be considered, such as chemicals, like chelating agents, leaching into the product, expansion of the containers in higher temperatures, and corrosion.[12]

There is very limited information on silicone packaging found in the literature. However, a review by Colas et al. briefly discusses how silicone tubing used in pharmaceutical applications can absorb ingredients with low molecular weight.[13] The chemical composition of the silicone tubing was not specified by the manufacturer, but this behavior aligns with the results found in this study. Further research needs to be conducted on the composition and properties of silicone packaging in a personal care context, especially considering that “TSA-approved” products have increased in popularity and are easily accessible to consumers.

METHODS

Stability testing. To evaluate stability, the sunscreens were transferred from their original packaging to alternative packaging and placed into 25°C and 45°C stability cabinets for 12 weeks. Each sample was tested for *in vitro* SPF, critical wavelength, viscosity, spreadability, pH, particle size, and aesthetics at weeks 0, 2, 4, 8, and 12. Additionally, each sunscreen underwent three freeze-thaw cycles in each packaging type.

There is currently no enforceable protocol on how to test the stability of sunscreens in the United States. The only requirement the FDA has for sunscreen stability is that they maintain a shelf-life of three years, but it is the manufacturer’s discretion on how to test this.[14] Guidelines provided by The European Cosmetic Toiletry and Perfumery Association (COLIPA), UL Prospector website, the International Council for Harmonisation of Technical Requirements for Pharmaceuticals for Human Use (ICH), and experts in the cosmetic industry influenced the design of this study.

In vitro SPF and critical wavelength testing. In vitro SPF and critical wavelength were determined based on the FDA 2011 method using the Labsphere 2000S (Labsphere, North Sutton, NH), described in detail in our previous work.[15] Two plates were scanned per sample, with a third scanned if the variability between the prior samples were too high.

Viscosity. Viscosity was tested in triplicates using a Brookfield DV-1 Prime Viscometer (Brookfield, Middleboro, MA) with spindle 27 and a small sample adapter at 25°C.

Spreadability. The spreadability of each sample was tested under compression on the TA.XT*Plus* Texture Analyzer (Texture Technologies, Hamilton, MA) using a TTC spreadability probe. The pre-test speed, test speed, and post-test speed were set to 3 mm/s, and the target mode was defined as distance of 11 mm. Approximately 8 g of each sunscreen was placed in the sample holder. The pre-set location was assigned at 6.5 mm. Each sunscreen sample was tested in triplicates at 25°C.

pH. The pH of each sample was determined using a pH meter (HANNA Instruments, Smithfield, RI) in triplicates at 25°C.

Particle size. Particle size of 30 particles from each sample was determined using a light microscope (Amscope, Irvine, CA) at weeks 0 and 12.

Evaluation of aesthetics. Aesthetics, including color, homogeneity, and signs of separation, were visually observed at the above-mentioned testing intervals, and photos were taken at weeks 0 and 12.

Freeze-thaw cycles. Each sample was placed in the freezer at -18°C for 24 hours, then thawed for 24 hours at 25°C to complete one cycle. Samples were observed for signs of separation.

Data analysis. Statistical analysis of *in vitro* SPF, critical wavelength, viscosity, spreadability, pH, and particle size were determined using one-way analysis of variance (ANOVA) followed by Tukey's multiple comparison test using SPSS Statistics 21 software (IBM, Armonk, NY). A p value less than 0.05 was taken as the minimal degree of statistical significance.

RESULTS AND DISCUSSION

IN VITRO SPF

SPF values measured during the 12-week period were compared to the baseline SPF and not the SPF claimed on the bottle. An important note is that it is not uncommon for *in vitro* SPF to not exactly match the claimed SPF on the bottle. Studies have shown that *in vitro* SPF values can be lower than what is claimed on the bottle.[16,17] Some factors that can influence *in vitro* results are the pressure applied to the polymethyl methacrylate (PMMA) plate, the speed used to spread the sample on the PMMA plate, the storage of the PMMA plates, or using multiple testers for the sunscreens throughout the study. Consistency is crucial for reliable and reproducible results when testing *in vitro*, and these were taken into consideration in this study by utilizing only one person to follow a standard method, storing the plates in controlled conditions, and spreading the sunscreens for the same period with consistent pressure.

Table I

In Vitro SPF Over the 12-Week Study

Zinc oxide-based sunscreen

Packaging	Week 0	Week 2	Week 4	Week 8	Week 12	Percent change from baseline (%)
25°C P	153.6 ± 8.4	139.7 ± 0.4	211.5 ± 19.7*	172.0 ± 16.5	162.0 ± 0.8	6
45°C P		176.2 ± 9.9	195.3 ± 29.2	151.9 ± 20.7	171.0 ± 23.1	11
25°C G		125.6 ± 0.0	139.1 ± 40.4	221.5 ± 20.2	162.0 ± 12.3	6
45°C G		123.9 ± 5.2	144.8 ± 101.0	201.8 ± 10.8	183.0 ± 65.2	19
25°C M		168.3 ± 33.7	133.8 ± 23.5	235.4 ± 15.7	164.0 ± 21.6	7
45°C M		174.7 ± 2.2	213.4 ± 32.1	291.5 ± 24.1*	274.0 ± 29.0*	78
25°C S		144.5 ± 27.0	229.4 ± 25.2	349.9 ± 17.1	343.0 ± 18.5	123
45°C S		383.1 ± 63.3*	325.7 ± 63.5	408.2 ± 57.7*	363.0 ± 36.1*	136

Titanium dioxide-based sunscreen

Packaging	Week 0	Week 2	Week 4	Week 8	Week 12	Percent change from baseline (%)
25°C P	225.8 ± 10.2	181.2 ± 21.3	273.8 ± 33.1	201.3 ± 25.6	365.0 ± 67.0	62
45°C P		182.4 ± 5.2	310.8 ± 25.9*	284.7 ± 10.3	319.0 ± 6.9*	41
25°C G		218.0 ± 9.9	328.1 ± 63.6	251.9 ± 23.0	368.0 ± 16.7	63
45°C G		169.9 ± 52.2	199.3 ± 48.2	324.3 ± 40.2	345.0 ± 26.8	53
25°C M		238.7 ± 13.5	256.7 ± 33.8	244.4 ± 0.9	354.0 ± 2.8*	57
45°C M		220.9 ± 18.8	248.5 ± 105.3	321.9 ± 13.5	374.0 ± 9.5	66
25°C S		N/A	N/A	N/A	N/A	–
45°C S		N/A	N/A	N/A	N/A	–

Combination sunscreen

Packaging	Week 0	Week 2	Week 4	Week 8	Week 12	Percent change from baseline (%)
25°C P	64.0 ± 1.3	60.8 ± 5.4	96.8 ± 8.3*	111.9 ± 5.5*	73.0 ± 5.5	14
45°C P		72.5 ± 1.7	40.4 ± 3.6	107.0 ± 22.1	128.0 ± 1.5*	100
25°C G		77.5 ± 9.0	65.9 ± 24.4	117.4 ± 3.3*	70.0 ± 8.0	9
45°C G		57.2 ± 18.4	71.6 ± 25.0	169.2 ± 20.1*	129.0 ± 16.0	102
25°C M		80.4 ± 9.9	120.7 ± 10.5*	134.8 ± 11.2*	120.0 ± 6.4*	88
45°C M		88.0 ± 15.2	57.8 ± 9.4	**	137.0 ± 4.6*	114
25°C S		86.3 ± 0.4	130.1 ± 26.2	376.3 ± 59.9*	471.0 ± 27.9*	636
45°C S		576.6 ± 32.7*	606.9 ± 19.1*	681.7 ± 83.2*	554.0 ± 35.2*	766

Notes: results are displayed as average ± SD.

*Significant change, p value < 0.05, **SPF could not be recorded at week 8 for the 45°C sample due to the container expanding and preventing opening. SPF at week 12 was recorded after breaking the container. N/A: products solidified and could no longer be tested.

The baseline SPF of the sunscreens in their original packaging was 153.6 ± 8.4 for ZnO, 225.8 ± 10.2 for TiO_2, and 64.0 ± 1.3 for the combination product (Table I). The SPF increased over the 12 weeks in all packaging to different extents, with the biggest change observed in the silicone packaging for all sunscreens. A significant difference from the baseline SPF measurement indicates that the product may no longer be as effective as advertised and is unstable. The SPF of the TiO_2 sunscreens was not recorded in silicone packaging after the baseline measurement because the samples solidified between weeks 0 and 2, preventing the proper spreading of the samples. This is an obvious indication of instability.

While the percent change in the packaging materials other than silicone is smaller, variation in SPF can question the product's integrity.

For a sunscreen to claim broad spectrum protection in the United States, it must have a critical wavelength of at least 370 nm. If the critical wavelength falls below 370 nm throughout its shelf-life, the consumer loses protection against UV radiation, and therefore the product is unstable. All three sunscreens in all types of packaging were consistently above 370 nm over the 12 weeks and were, therefore, stable from a broad-spectrum protection perspective.

VISCOSITY

Sunscreen emulsions typically display shear-thinning flow behavior, where the viscosity decreases as the shear rate increases. A significant increase or decrease in viscosity over time indicates instability and can contribute to its inability to spread or stay on the skin.

The viscosity of the ZnO, TiO_2, and combination sunscreens in the plastic, glass, and metal containers displayed shear-thinning behavior throughout the testing period (Figure S1). Samples in the silicone containers were not measured due to product consistency changes and/or leakage. The viscosity of all samples at 45°C decreased significantly ($p < 0.05$); however, this is not concerning considering that matter flows faster at higher temperatures.

SPREADABILITY

The ability to easily spread a product on the skin is another attribute consumers consider when purchasing sunscreen. When measuring spreadability, four parameters are analyzed: firmness, hardness, stickiness, and adhesiveness. A product on the market should be able to maintain these four properties throughout its shelf life, and significant changes in spreadability can directly affect the consumer's ability to use a product properly.

In general, firmness and hardness work done increased for all sunscreens over the 12 weeks at both temperatures (Tables II). Stickiness also increased by the end of the testing period at both temperatures. Sunscreen transferred into the silicone packaging had the greatest change in overall spreadability values. Samples at both 25°C and 45°C solidified before the end of the 12-week testing period, making the products not spreadable and unstable. Additionally, it is important to note that spreadability in the silicone bottles could not be measured past week 4 at 25°C and week 2 at 45°C as the formulations were leaking from its packaging. This occurrence, in addition to the spreadability results, deem silicone an unfit packaging type from a spreadability perspective.

PH

pH was measured to evaluate any chemical changes occurring in the sunscreens. It is typical for mineral sunscreens to have a pH of 7–8.[18] Below pH of approximately 6, Zn^{2+} ions can begin to migrate in ZnO-containing formulations, potentially ruining the formulation.[18]

The initial pH of each sunscreen was slightly acidic, with the ZnO sunscreen at 6.62 ± 0.08, the TiO_2 sunscreen at 6.33 ± 0.19, and the combination at 6.95 ± 0.16. pH of the sunscreens stayed in the range of approximately 6–8 over the 12 weeks; more specifically, the ZnO sunscreens' pH was in the range of 5.92 to 7.73 across different packaging types, the TiO_2 sunscreens in the range of 5.33 and 7.38 across different packaging types, and in the range of 6.09 and 7.01 for the combination sunscreens across different packaging types. All sunscreens were considered stable during the 12-week stability study.

PARTICLE SIZE

Measuring the particle size of the UV filters gives an indication of a sunscreen's stability from a uniformity perspective. A significant increase in particle size indicates that agglomeration is occurring, which can affect its extent of protection, the density of the phase the inorganic UV filter resides in, and stability of the emulsion.

The baseline particle size was 0.23 ± 0.05 μm for the ZnO-based, 0.40 ± 0.25 μm for the TiO_2-based, and 0.27 ± 0.12 μm for the combination sunscreens. The particle size of the UV filters in the ZnO and combination sunscreens remained uniform in all packaging types throughout the 12 weeks (Table SII). In the TiO_2-based sunscreens, the particle size in plastic packaging at 45°C and glass packaging at 25°C increased significantly to 1.19 ± 1.21 μm and 1.36 ± 0.64 μm, respectively. Particle size in silicone packaging was not measured due to its solidification after 2 weeks.

AESTHETICS

In addition to claims, how a sunscreen looks and smells are other factors that consumers consider when purchasing a product. A sunscreen should maintain the same color and smell throughout its shelf-life. Changes in these factors are signs of emulsion instability or a lack of preservative efficacy, and therefore are unstable.

At week 0, the ZnO sunscreen had an off-white, eggshell color (Figure 1). After 12 weeks, the plastic, glass, and metal samples at 25°C showed no changes aesthetically and were stable. However, the plastic, glass, and metal samples at 45°C displayed varying levels of separation where a yellow, transparent liquid was floating on the top of the sample in the containers. This is an indication of creaming, and these samples were considered unstable. The samples in the silicone packaging at both 25°C and 45°C changed significantly, where both samples deepened in color and were visibly thicker.

At week 0, the TiO_2 sunscreen was bright white and shiny (Figure 1). After 12 weeks, the plastic, glass, and metal samples at 25°C and the plastic sample at 45°C showed no changes aesthetically; however, they decreased in viscosity. The glass and metal samples at 45°C displayed slight separation where a colorless, transparent liquid was floating on the top of the samples and were considered unstable. The samples in the silicone packaging at both 25°C and 45°C changed significantly after two weeks. The sample at 25 °C solidified to soft

Table II

Spreadability of Sunscreen in Plastic Packaging

Zinc oxide-based sunscreen

		Time point	Firmness (g)	Hardness work done (g.sec)	Stickiness (g)	Adhesiveness (g.sec)
		Baseline	8.62 ± 0.15	8.96 ± 1.14	−5.93 ± 0.20	−0.08 ± 0.01
25°C	P	12 weeks	9.49 ± 0.86	9.56 ± 0.17	−6.68 ± 1.20	−0.08 ± 0.00
	G	12 weeks	10.43 ± 0.35	9.73 ± 0.36	−7.40 ± 0.23	−0.08 ± 0.00
	M	12 weeks	9.23 ± 0.50	9.02 ± 0.23	−7.17 ± 0.76	−0.08 ± 0.00
	S	4 weeks	71.91 ± 5.96*	42.27 ± 3.26*	−53.83 ± 3.51*	−84.51 ± 2.82*
45°C	P	12 weeks	9.08 ± 3.01	9.60 ± 0.62	−5.98 ± 2.42	−0.06 ± 0.04
	G	12 weeks	7.45 ± 0.71	9.51 ± 0.180	−5.52 ± 0.95	−0.08 ± 0.00
	M	12 weeks	7.45 ± 1.10	9.94 ± 0.69	−5.12 ± 1.65	−0.09 ± 0.00
	S	2 weeks	29.2 ± 4.76*	15.00 ± 2.02	−13.48 ± 2.23*	−28.81 ± 2.99*

TiO$_2$-based sunscreen

		Time point	Firmness (g)	Hardness work done (g.sec)	Stickiness (g)	Adhesiveness (g.sec)
		Baseline	5.97 ± 0.20	8.28 ± 0.34	−4.14 ± 0.23	−0.08 ± 0.00
25°C	P	12 weeks	7.10 ± 1.56	9.40 ± 0.66	−3.70 ± 3.18	−0.08 ± 0.00
	G	12 weeks	8.27 ± 1.15	9.31 ± 0.27	−5.62 ± 0.75	−0.08 ± 0.00
	M	12 weeks	6.23 ± 0.16	9.79 ± 0.49*	−2.87 ± 2.43	−0.08 ± 0.00*
	S	-	N/A	N/A	N/A	N/A
45°C	P	12 weeks	8.66 ± 0.06	9.96 ± 0.16	−7.07 ± 0.64	−0.08 ± 0.00
	G	12 weeks	13.05 ± 0.15*	9.96 ± 0.23*	−8.54 ± 0.59*	−0.08 ± 0.00*
	M	12 weeks	10.92 ± 0.44*	9.85 ± 0.25	−6.81 ± 0.87	−0.08 ± 0.00*
	S	–	N/A	N/A	N/A	N/A

N/A: products solidified and could no longer be tested.

Combination sunscreen

		Time point	Firmness (g)	Hardness work done (g.sec)	Stickiness (g)	Adhesiveness (g.sec)
		Baseline	12.84 ± 0.50	7.91 ± 0.29	−8.57 ± 0.48	−0.08 ± 0.00
25°C	P	12 weeks	12.40 ± 1.71	9.59 ± 0.03*	−8.02 ± 0.79	−0.08 ± 0.00
	G	12 weeks	11.71 ± 1.30	9.61 ± 0.27*	−7.41 ± 0.87	−0.08 ± 0.00*
	M	12 weeks	14.70 ± 1.91	9.34 ± 0.26*	−9.53 ± 0.54	−0.08 ± 0.00*
	S	4 weeks	22.08 ± 2.38*	13.68 ± 1.17*	−15.33 ± 2.15*	−17.62 ± 2.60*
45°C	P	12 weeks	12.55 ± 4.21	9.29 ± 1.11	−7.89 ± 3.25	−0.08 ± 0.01
	G	12 weeks	7.98 ± 2.38*	9.60 ± 0.11*	−4.91 ± 1.94	−0.09 ± 0.00*
	M	12 weeks	15.24 ± 2.52	9.72 ± 0.28*	−7.75 ± 1.02	−0.08 ± 0.00*
	S	2 weeks	8.74 ± 1.41	11.57 ± 0.04*	−3.72 ± 2.63*	−0.04 ± 0.00*

Note: Results displayed as average ± SD.
*Significant change, p value < 0.05.

white, crumbly curdles while the sample at 45°C solidified to brittle, light brown plates. This was an obvious sign of instability.

At week 0, the combination sunscreen was bright white and shiny (Figure 1). After 12 weeks, the plastic, glass, and metal samples at 25°C and the metal sample at 45°C remained

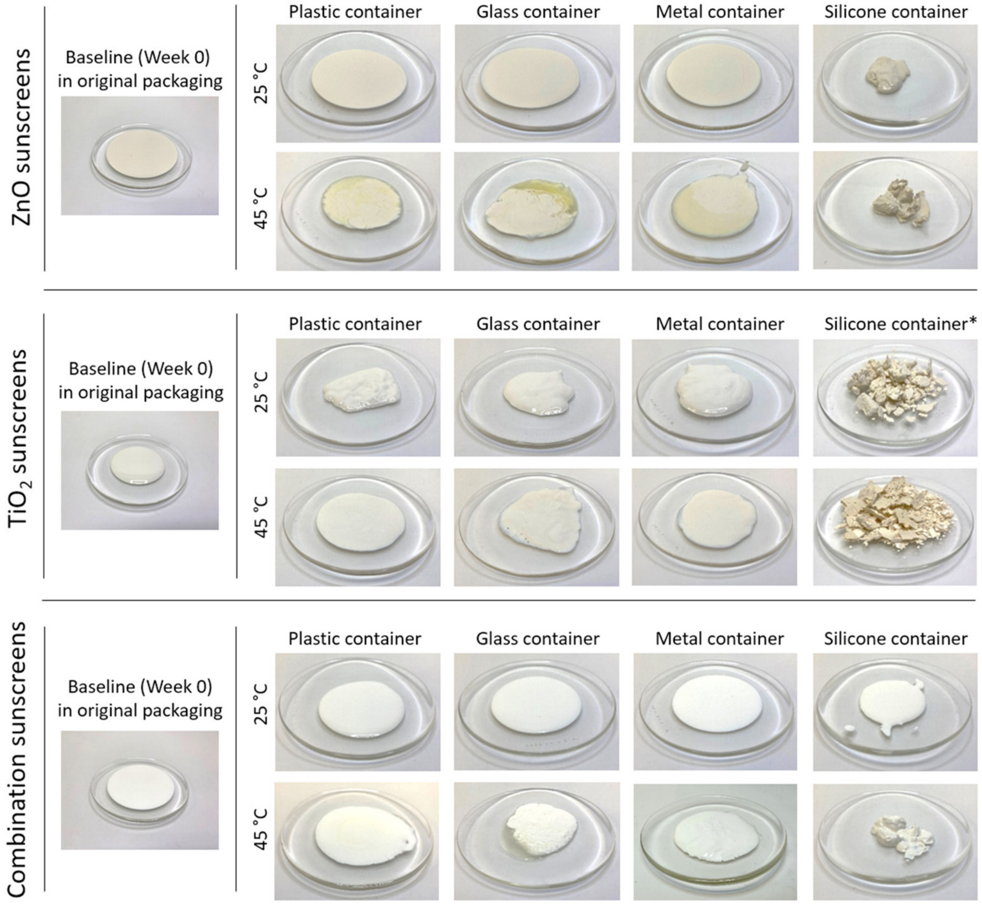

Figure 1. Sunscreens' appearance at week 12, *week 2 for the silicone container with the TiO_2 sunscreen.

aesthetically consistent and stable. However, the plastic and glass samples at 45°C displayed varying amounts of separation where a mix of a colorless and yellow transparent liquid was present on the top of the samples. These samples were considered unstable. The samples in the silicone packaging at both 25°C and 45°C changed significantly; the room temperature sample slightly thickened and was excreting oils from its packaging, and the sample at 45°C became visibly thicker. These changes also indicated instability and would have a negative impact on a consumer's experience.

FREEZE-THAW CYCLES

Conducting freeze-thaw cycles indicates how a product can withstand extreme temperature changes and is used as a quick indicator of product stability. This is especially important when traveling, for example, as airplanes fly to altitudes with frigid temperatures. Sunscreen separation is an indication of instability.

The TiO_2 and combination sunscreens were stable, as they did not show any change in any packaging type after three freeze-thaw cycles. The ZnO sunscreen, however, separated in all packaging types, including in its original packaging. It can be concluded that the formulation itself cannot withstand extreme temperature changes regardless of the type of packaging it is in. Stability from a packaging perspective for the ZnO-based sunscreen cannot be commented on in this study.

CONCLUSIONS

The goal of this study was to determine the stability of inorganic sunscreen emulsions when transferring them to alternative packaging. Ideally, sunscreens should maintain consistent SPF, critical wavelength, viscosity, spreadability, pH, particle size, and aesthetics, and should remain stable under extreme temperatures.

This study is the first study to our knowledge that focused on inorganic sunscreens. Prior research evaluated the stability of chemical UV filters in plastic, glass, and metal packaging, but not inorganic sunscreens or with the use of silicone packaging.[19]

This study confirms that using alternative packaging to store sunscreens negatively impacts the stability of the formulation. While transferring products to travel-size containers may be a trendier or more convenient option, *in vitro* SPF, spreadability, particle size, and data collected visually clearly displays that all alternative packaging types tested, especially silicone packaging, will change the extent of the product's protection, its ability to spread, and its homogeneity on a visual and molecular level. It is likely that the silicone packaging was permeable to some of the sunscreens' ingredients, either causing leakage or solidification of the leftover material contained inside them. It is also possible that ingredients within the sunscreen formulations could have partially dissolved the packaging, causing those chemicals to become introduced to the formulation, or that some ingredients had evaporated in the packaging, causing parts of the formulation to become more concentrated.

It is recommended that consumers purchase travel-sized sunscreens rather than transferring their products to alternative packaging to maintain the integrity of the product. Companies that sell travel-sized sunscreens run stability studies that must meet desired specifications in the original packaging before being sold on the shelves, leaving the consumer with no doubt about the reliability to protect themselves from UV radiation.

We hope that the results of this study can guide consumers who are not aware that in addition to the interactions of raw materials in a formulation, packaging type can also impact stability. The results of this study can help deter consumers from transferring their personal care products into alternative packaging when traveling.

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SUPPORTING INFORMATION

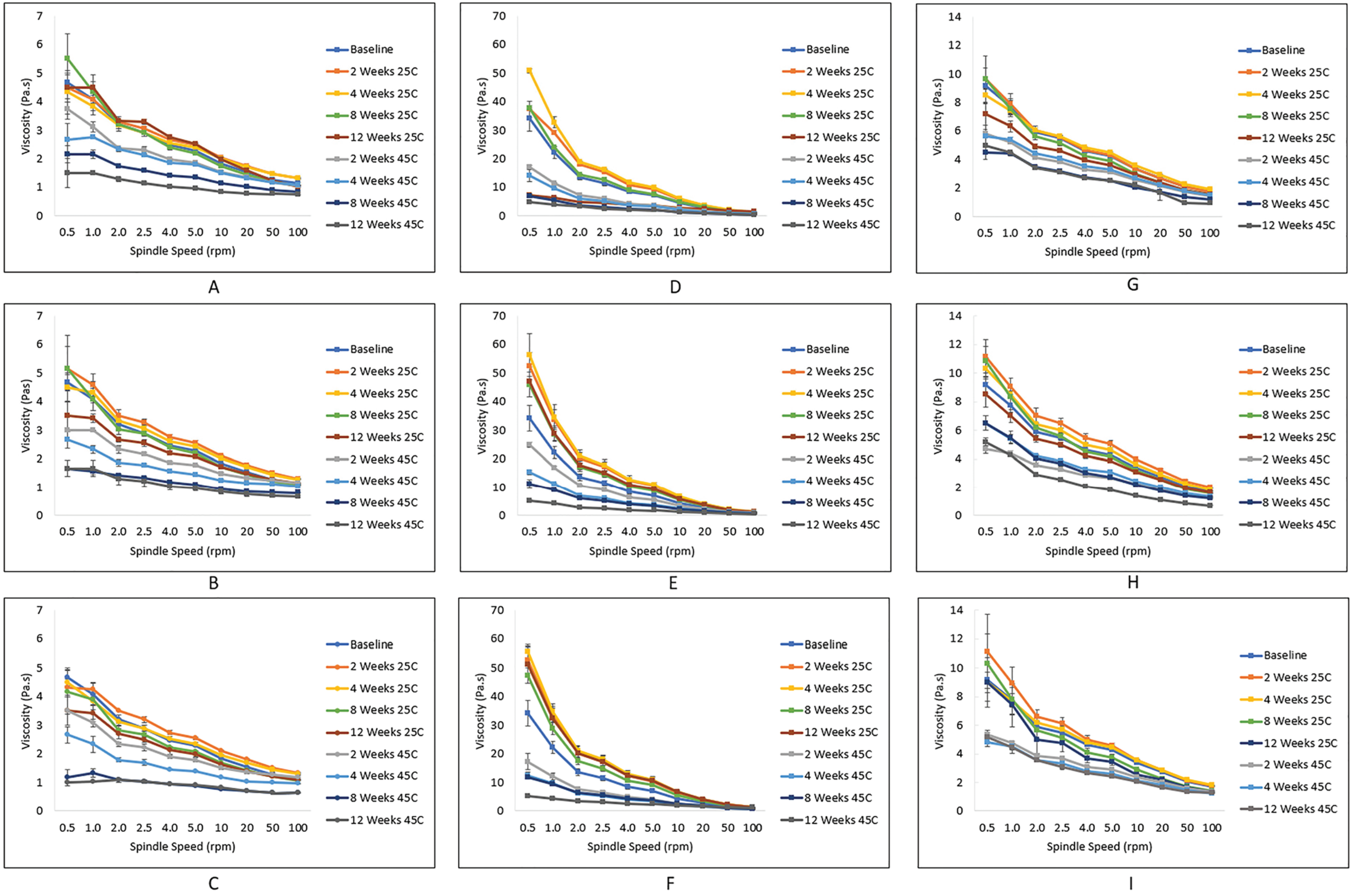

Figure S1. Viscosity of sunscreens. (A) ZnO in plastic, (B) ZnO in glass, (C) ZnO in metal packaging; (D) TiO_2 in plastic, (E) TiO_2 in glass, (F) TiO_2 in metal packaging; (G) combination in plastic, (H) combination in glass, (I) combination in metal packaging.

Table SI

Ingredients in the Sunscreens Used in This Study

Sunscreen	Ingredients (INCI names)
Coppertone® Pure and Simple Kids SPF 50	Active ingredient(s): zinc oxide (24.08%) Inactive ingredients: water, C12-15 alkyl benzoate, isopropyl palmitate, butyloctyl salicylate, ethylhexyl isononanoate, cetyl PEG/PPG-10/1 dimethicone, propylene glycol, cyclopentasiloxane, bis-ocyldodecyl dimer dilinoleate/propanediol copolymer, dimethicone, ethylhexyl methoxycrylene, polyester-27, tea (camellia sinesis) leaf extract, Giant Kelp (macrocytsis pyrifera) extract, Sacred Lotus (nelumbo nucifera) extract, triethoxycaprylylsilane, beeswax, hydroxyacetophenone, PEG-12 dimethicone crosspolymer, tocopherol, 1,2-hexanediol, caprylyl glycol, sodium chloride
Ombrelle® Kids Sunscreen Lotion SPF 50+	Active ingredient(s): titanium dioxide (15%) Inactive ingredients: water, dimethicone, C12-15 alkyl benzoate, isohexadecane, talc, styrene/acrylates copolymer, isododecane, caprylyl methicone, dicaprylyl ether, triethylhexanoin, dicaprylyl carbonate, dimethicone/PEG-10/15 crosspolymer, aluminum hydroxide, stearic acid, pentylene glycol, DL-alpha-tocopherol, phenoxyethanol, PEG-9 polydimethylsiloxyethyl dimethicone, magnesium sulfate, PEG-8 laurate, PEG-9, polyhydroxystearic acid, propylene carbonate, caprylyl glycol, disteardimonium hectorite, aluminum stearate, alumina
Banana Boat® Sport Sunscreen Lotion SPF 50+	Active ingredient(s): titanium dioxide (4.50%), zinc oxide (6.50%) Inactive ingredients: water, caprylic/capric triglyceride, isohexadecane, butyloctyl salicylate, octyldodecyl citrate crosspolymer, cetyl PEG/PPG-10/1 dimethicone, lauryl PEG-8 dimethicone, C30-38 olefin/isopropyl maleate/MA crosspolymer, sodium chloride, ethylhexyl methoxycrylene, dimethicone, phenoxyethanol, caprylyl glycol, PEG-8, alumina, glycerin, sodium citrate, tocopheryl acetate

Table SII
Average Particle Size of UV Filters in μm

Sunscreen Type	Plastic at week 12		Glass at week 12		Metal at week 12		Silicone at week 12	
	25°C	45°C	25°C	45°C	25°C	45°C	25°C	45°C
ZnO	0.29 ± 0.09	0.24 ± 0.06	0.21 ± 0.07	0.27 ± 0.08	0.28 ± 0.1	0.27 ± 0.09	0.30 ± 0.14	0.28 ± 0.14
TiO_2	0.31 ± 0.16	1.19 ± 1.21*	1.36 ± 0.64*	1.01 ± 1.47	0.91 ± 0.66	0.67 ± 0.32	N/A	N/A
Combination	0.21 ± 0.05	0.29 ± 0.11	0.34 ± 0.17	0.32 ± 0.19	0.38 ± 0.19	0.21 ± 0.06	0.21 ± 0.06	0.30 ± 0.12

Note: Results displayed as average ± SD.