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Bridging personal care and environmental care: A holistic approach to formulation development in the post-COVID-19 era

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Introduction of research

Despite the global disruption triggered by the COVID-19 pandemic and the ensuing supply chain interruptions that persist, the pursuit of sustainability remains a dominant megatrend throughout society. In the cosmetic industry, a key sustainability objective is to provide consumers with improved personal care products that simultaneously reduce environmental impact while meeting current skin care needs, especially for cleansing and therapeutic benefits.

This presentation will demonstrate formulation strategies that employ ingredients with improved sustainability profiles to deliver products that meet consumer expectations for mildness, efficacy, and sensory experience. Polyesteramines (PEAs), e.g. Polyester-11 and -37, exhibit lower environmental risk compared to traditional conditioning polymers due to their non-quaternary ammonium cationic groups and ready biodegradability. The use of polyesteramines in surfactant-based cleansers is shown to dramatically improve the skin mildness of relatively harsh commodity surfactants and provides the benefits of synergistic viscosity building and easy-rinsing skin conditioning. Amino lipids, e.g. Brassicyl Valinate Esylate (BVE), also demonstrate superior environmental profiles relative to conventional quaternary ammonium compounds, such as Distearyldimonium Chloride, yet they deliver equivalent performance for formulating lamellar liquid crystalline emulsions for highly efficacious moisturizers.

Polyesteramines

Cleansers are an important tool for preventing the spread of infectious disease and improving overall health and wellbeing. Surfactants are the key cleansing ingredient in these formulations, but their chronic usage can result in dryness, redness, swelling, and sensorial irritation.[7] Thus, formulators are challenged to make cleansing products that are efficacious at removing dirt and germs and also provide consumer preferred claims of gentleness, e.g. nondrying. One way to achieve this is by using surface-active polymers that strongly interact with typically harsh anionic surfactants to sequester the excess surfactant monomers and micelles making them less available to penetrate the skin or remove skin barrier components.[3]

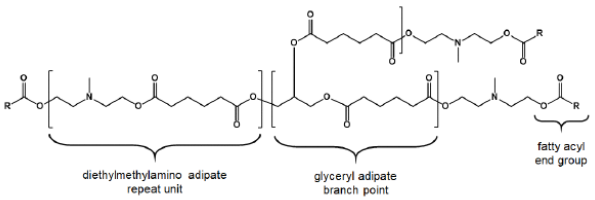

Figure 1. Chemical structures of PE-11 and PE-37 polyesteramines.

Polyesteramines PE-11 and PE-37 are surface-active copolymers of adipic acid, bis-(hydroxyethyl)methylamine, glycerin, and fatty acids. They are partially biobased, derived from coconut and palm oil, and readily biodegradable due to the ester linkages in the backbone, which improves their sustainability profile relative to traditional cationic conditioning polymers. The tertiary

amino groups are pH-responsive and can be rendered cationic by protonation in water at acidic pH. Protonated PEAs specifically adsorb anionic surfactants by electrostatic interactions and nonspecifically adsorb all surfactants by hydrophobic interactions of the fatty acyl end groups with the surfactant hydrophobic tails.

In a surfactant system of 10 wt% of sodium alpha olefin sulfonate and cocamidopropyl betaine (AOS/CAPB) in a 3:1 ratio, the mildness enhancement of PE-11 and PE-37 was compared with polyquaternium-7 (PQ-7), a traditional cationic conditioning polymer. PQ-7 is a hydrophilic polymer that has a permanent cationic charge which presents a strong potential for environmental persistence and aquatic toxicity. Irritation mitigation was quantified by the change in critical micelle concentration (ΔCMC) of the surfactant system in presence of the polymers and by in-vitro irritation studies.

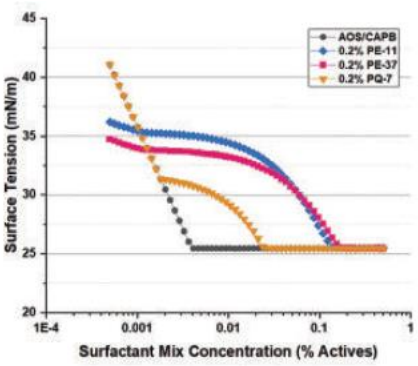

Figure 2. Equilibrium surface tensiometry plots for AOS/CAPB alone and in the presence of the compared polymers.

The ΔCMC of the surfactant system alone and in presence of a polymer is a measure of surfactant binding, whereas the larger ΔCMC the greater, the surfactant binding. The two PEAs have an initial lower surface tension value that demonstrates the inherent surface activity (PE-11 = 36.7 mN/m, PE-37 = 35.2 mN/m), compared to PQ-7. The PEAs demonstrate a greater change in critical micelle concentration compared to PQ-7, which shows minimal surfactant binding capacity (Figure 3).

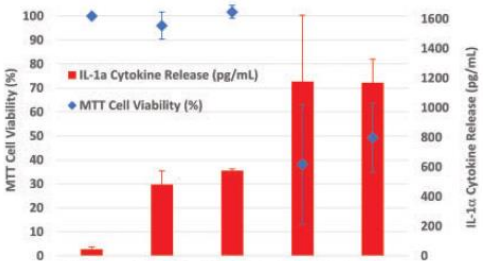

Figure 3. MTT cell viability and IL-1α cytokine release data for the AOS/CAPB without and with 2% of the compared polymers.

To quantify skin irritation, in-vitro EpiDerm™ studies were done with endpoints of MTT cell viability and IL-1α cytokine release. Both endpoints have been shown to have correlation to clinical measures of irritation such as expert grading and transepidermal water loss.[2] The irritation potential in terms of both MTT cell viability and IL-1α cytokine release was dramatically reduced with 2% PEA when compared to the formulas without polymer and with 2% PQ-7.

Brassicyl Valinate Esylate (BVE)

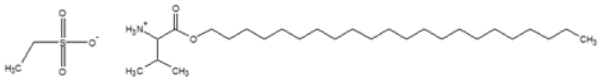

Figure 4. Chemical structure of BVE.

Therapeutic lotions are recommended to reduce further irritation and dryness that result from the removal of skin barrier components after cleanser use. Efficacious moisturizing lotions should contain high amounts of emollients, humectants, and occlusives, yet must also have a consumer preferred sensory and sustainability profile.[5] Formulating with all these ingredients could potentially yield greasy and tacky aesthetics if using traditional nonionic emulsifiers. Cationic emulsifiers in combination with fatty alcohols form lamellar liquid crystal (LLC) structures that are viscous, shear-thinning, and have a desirable feel and slip. The LLC formulation strategy is employed for example in the AVEENO® Daily Moisturizing Lotion (DML)[6]. Amino lipids, such as BVE, are cationic emulsifiers capable of forming LLCs, yet have a better environmental profile compared to conventional quats.

Item	Ingredients (INCI)	Formula Amount (wt%)
1	Deionized Water	70.90
2	Sodium Chloride	0.01
3	Calcium Gluconate	0.84
4	Glycerin	12.00
5	Colloidal Oatmeal	1.00
6	**Brassica Alcohol (and) Brassica Valinate Esylate (and) Brassica Glycerides**	**6.00**
7	**Isopropyl Palmitate**	**3.00**
8	Petrolatum	4.00
9	Dimethicone	1.25
10	**Caprylhydroxamic Acid (and) Benzyl Alcohol (and) Glycerin**	**1.00**
		100.00

Figure 5. BVE in a DML-type formulation.

BVE can be employed in a similar formulation that contains high levels of glycerin, isopropyl palmitate, and petrolatum, as shown in Figure 5, and achieve LLC structure formation. In Figure 6, the birefringence under polarized light microscopy showed Maltese crosses indicative of LLCs for both the BVE formulation and AVEENO® DML that contains petro-derived Distearyldimonium Chloride. BVE differs in that it is 100% biobased-based, comprised of valine derived from the biofermentation of sugarcane glucose, brassica rapa seed oil, and ethane sulfonic acid derived from biobased ethanol. Distearyldimonium Chloride is categorized as a skin corrosion/irritation hazard and as an environmental hazard due to its quaternary ammonium functionality.

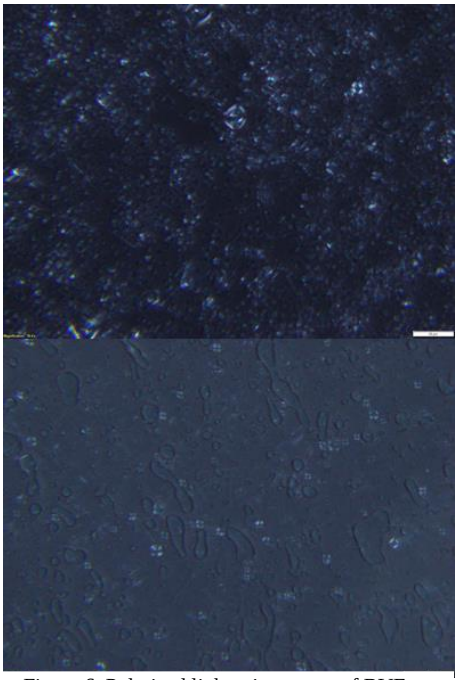

Figure 6. Polarized light microscopy of BVE DML type formula (top) and Aveeno® DML (bottom).

Conclusion

Understanding the different ingredients that go into cleansing and therapeutic moisturizing formulations allows us to maintain the efficacy of the formula while making improvements in mildness and sustainability without compromising performance. Polyesteramines in cleansing formulas improve both mildness and sustainability in harsh commodity surfactant systems, and 100% biobased amino lipids can be used for maintaining a desirable sensory profile in high emollient-high humectant lotion formulations via LLC structure formation.

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About the speaker

Brittany Pease is an Application Scientist II for the Formulation, Technology, and Innovation team at INOLEX, a leading global specialty ingredients company headquartered in Philadelphia, Pennsylvania with locations in ten countries. Previously, Brittany worked as Applications Chemist for INOLEX's former industrial lubricants division, LEXOLUBE. Prior to working at INOLEX, she worked for an industrial lubricant testing laboratory, Petro-Lubricant Testing Laboratories Inc., and worked product support for a medical device company, Instrumentation Laboratory, A Werfen Company. Brittany earned her M.S. in Cosmetic Sciences at the James L. Winkle College of Pharmacy in University of Cincinnati in 2021 and earned her B.S. in Chemistry at The College of New Jersey in 2014.

Qualitative and quantitative interactions of cationic surfactants with cosmetic anionic ingredients

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Introduction of research

Surfactants are known for their detergency properties and are part of many cosmetic applications. On top of this characteristic displayed by anionic surfactants, cationic surfactants on the other hand are also used as broad-spectrum antimicrobial agents.

There is a well spread belief that anionic species would systematically interact with cationic surfactants, thus inhibiting their efficiency and leading to some sort of automatic incompatibility. The present study aims at revisiting this belief using scientific tools targeting the establishment of objective conclusions.

Isothermal Titration Calorimetry (ITC) is a well-known technique in the biomolecular field, that quantitatively determine the thermodynamic parameter of molecular interactions [1-2]. Conductimetric methods have been used to characterize ionic surfactants behavior in solution [3].

The research purpose was to develop a methodology using physico-chemical tools to determine the degree of interactions between cationic preservative species and anionic compounds and to achieve a proper selection of cationic preservative to a given cosmetic formulation, in presence of anionic surfactants.

Results and discussion

The molecular interactions were investigated by monitoring the conductivity of 1:1 molar ratio of Didecyldimethylammonium chloride (DDAC) and anionic surfactants. Figure 1 shows the 'measured to theoretical' conductivity ratio for each pair. Increasing the concentration of the molecules increased the 'measured to theoretical' conductivity ratio. At 10mM of each molecule the majority of the 'measured to theoretical' ratio values exceeded 100%. The conductance behavior of the combined molecules (*i.e.*, measured) is expected to differ from the sum of the individual component solution's (*i.e.*, theoretical) once molecular interactions molecules take place. The stronger the molecular interactions are, the larger the deviation is expected to be.

Mixtures of DDAC and anionic molecules having sulfate or sulfonate functional group exhibited conductivity profiles with higher slopes compared to DDAC and carboxylate pairs.

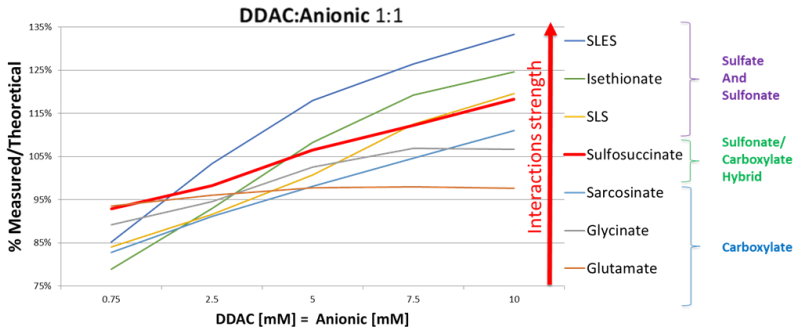

Figure 1: 'Measured to theoretical' conductivity ratio for each DDAC:anionic surfactant pair (1:1 molar ratio) at the concentration range of 0.75-10mM (at 25°C).

The molecular interactions between the cationic DDAC and various anionic molecules were further detected by ITC. The binding process could be directly measured by monitoring the heat evolution due to the molecular interactions, thereby determining the values of the binding constant (Ka), the stoichiometry (n) and the enthalpy of binding (ΔH) [2]. The integrated data of the enthalpy variation (ΔH) per mole of DDAC are plotted *vs.* the DDAC/anionic surfactant molar ratio (Figure 2). The measurements indicate a distinct difference in the behavior of anionic surfactants having a sulfate or sulfonate group when mixed with DDAC (higher enthalpy changes) in comparison with anionic surfactants bearing a carboxylate group.

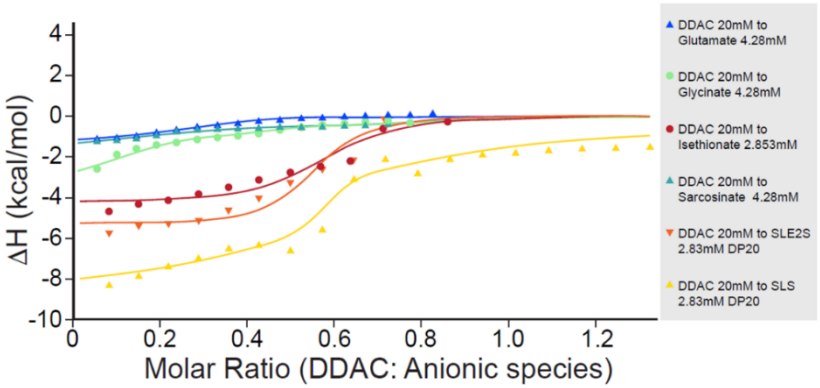

Figure 2: Enthalpy change per injection *vs.* DDAC/anionic surfactant molar ratio. DDAC was injected into a reaction cell containing different aqueous anionic solutions at RT.

In order to corroborate the above results with the antimicrobial efficacy of cationic surfactants, the challenge tests shown in Table 1 were performed, with the cationic preservative surfactant (0.5% of the total final product) within the designated cosmetic anionic shampoo.

Two pairs of anionic and cationic surfactants were selected, SLES/DDAC and Glutamate/Polyquaternium-80, representing the strongest and weakest interactions. Strong ionic interactions of sulfate-based molecules and DDAC led to a failed challenge test, while weak interactions between carboxylate-based molecules and P-80 did not interfere with the cationic antimicrobial activity which resulted in passing the challenge test.

Composition	Time	*E.Coli* (Gram- Bacteria)	*C. albicans* (Yeast)
Anionic surfactant in Shampoo: SLES Cationic surfactant in preservative: DDAC	Inoculum	1.1x10^6	1x10^5
	2 days	2x10^5	1x10^5
	7 days	2x10^5	1.8x10^3
	14 days	2x10^5	2.5x10^2
	21 days	2x10^5	<10
	28 days	2.6x10^5	<10

Composition	Time	*E.Coli* (Gram- Bacteria)	*C. albicans* (Yeast)
Anionic surfactant in Shampoo: Glutamate Cationic surfactant in preservative: P-80	Inoculum	1.1x10^6	1x10^5
	2 days	<10	<10
	7 days	<10	<10
	14 days	<10	<10
	21 days	<10	<10
	28 days	<10	<10

Table 1: Challenge tests of 0.5% preservative mixtures containing DDAC or Polyquaternium-80 in anionic shampoo cosmetic formulation containing SLES or Glutamate, respectively.

Conclusion

This study reveals that far to be a systematic phenomenon, not all anionic species would interact with cationic surfactants but only a specific subgroup. The tools developed allow to quickly assess and predict the interactions between any anionic and cationic species with reliability.

This innovative work opens a new window of opportunities for ingredients selection based on the scientific understanding of the molecular interactions.

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About the speaker

Paul Salama, Ph.D, Eng.

CTO & Head of Innovation

Dr. Paul Salama holds a B.Sc. in Chemistry and a M.Sc. in Chemical Engineering both from University of Lyon (France), along with a Ph.D. in Organic Chemistry from University of Montreal (Canada).

After having started his career as a university professor in Canada, he moved to the pharmaceutical industry in Canada then in Israel, holding senior research positions (VP CMC).

Dr. Salama leads the R&D activities of Sharon Laboratories since 2014 as its CTO and Head of Innovation.

Assessing baby cleanser mildness: combining non-invasive testing on adults and predictive computational modeling of the infant epidermis

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Introduction of research

Healthy infant skin depends upon the presence of a healthy skin barrier, which performs essential functions of retaining water to maintain proper hydration, inhibiting the penetration of microbes and/or allergens, thermoregulation, and gas exchange [1]. Infant skin differs from adult skin in several ways [2]. The stratum corneum is approximately 30% thinner in infants than in adults, and infant corneocytes and granular cells are smaller and exhibit more rapid turnover than those in adults [3]. Barrier properties of the stratum corneum, including water holding and transport, differ between adults and infants/children [4,5]. Water diffusion profiles for infants show a lower resistance to water flow throughout the stratum corneum compared with adults, corresponding to a less efficient barrier function [6].

Since infant skin is different and is still developing, it requires specially formulated, mild skincare products that should not disrupt the skin barrier [1]. Certain surfactants in skin cleansers may be harsh and damage the infant skin barrier and lead to irritation. Thus, measuring the effectiveness of the infant skin barrier is important in assessing the mildness of products for use on infant skin. However, there are concerns with testing on infants that may preclude or limit the use of certain methods and/or the numbers of participants. A novel method of assessing product mildness in adults was developed involving comparing the amount of penetration of a topically applied marker before and after product treatment (patch) with non-invasive confocal Raman microspectroscopy. This method gives results consistent with the exaggerated patch test method in adults [7]. Finally, a validated computational model of the infant epidermis can use adult data to predict the penetration of the marker in infant skin [8]. Here, this process is applied for the first time to predict the mildness of cleanser products on infant skin, using clinical data of marker penetration in adults.

Methodology

Eleven healthy participants aged 20-40 years with Fitzpatrick skin type II-IV participated in the study. Exclusion criteria included having a pre-existing dermatologic or chronic medical condition that could interfere with the study or pose a health risk to the participant. Each participant acclimated in a temperature- and humidity-controlled room for 15 min prior to baseline skin measurements with Raman spectroscopy (Skin Analyzer Model 3510, RiverD International B.V.). Next, a patch containing a freshly made dilution of cleanser product in deionized water was applied at each skin site. After 3 hours, the patches were removed, and after 20 min, a patch with freshly made 1.8% caffeine was applied to each skin site. After 30 min, the patch was removed, and final measurements with Raman spectroscopy were obtained as previously described [8]. Spectral analysis was performed using a customized algorithm for the Skin Tools software. Data from experimental caffeine penetration profiles were transferred to a

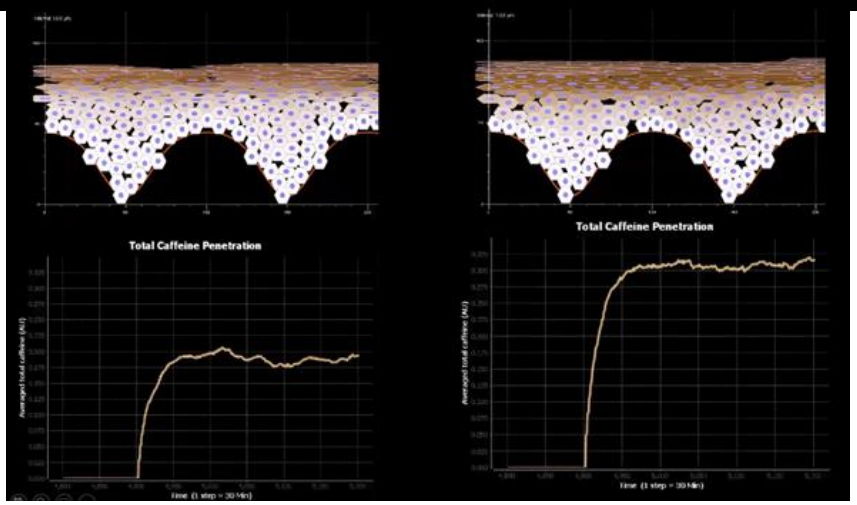

Figure 1. Comparison of caffeine penetration of the skin following application of two different products.

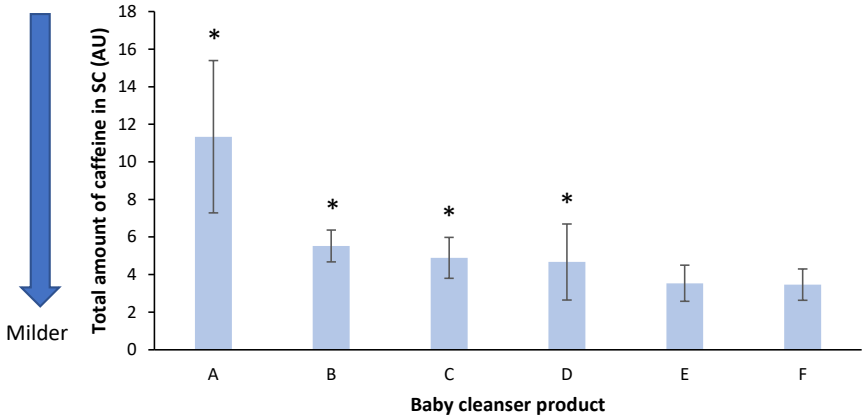

Figure 2. Predicted caffeine penetration in the stratum corneum of cleanser products in an infant skin model. Error bars show standard deviation. SC, stratum corneum.

* $p<0.05$ vs Product F.

validated computational model of infant epidermis that accounts for the unique structural and functional properties of infant skin [8]. The amount of caffeine penetrating in infant skin as predicted by the model was then calculated and data were analyzed via Student's t-test.

Results/Discussion

Six different cleanser products were clinically tested on the participants' volar forearms, followed by the caffeine permeability test and measurement via Raman microspectroscopy. These data were entered into the adult computational model for caffeine penetration. Representative data for caffeine penetration following application of cleanser products are shown in **Figure 1**. Results were then related to their predicted effect on infant skin, with the amount of caffeine predicted by the computational model to penetrate the infant stratum corneum following application of each product (**Figure 2**). Less predicted caffeine penetration corresponds to less barrier damage and thus a milder product, so Products E and F are the mildest, with Products A-D demonstrating significantly higher caffeine penetration.

Conclusion

This validated novel infant computational model enables adult testing of baby cleansers to become relevant to infant skin, allowing the relative ease of product testing on adults rather than on infants. The method takes less time compared to other exaggerated tests, is non-invasive, and shows a better dynamic range to distinguish among formulations, particularly mild formulations, compared with other accepted models. It also confers the ability to screen for effects of products on the skin that may not be apparent from the product ingredients.

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About the speaker

Georgios Stamatas, PhD, is a biomedical engineer with more than 20 years of international experience in the health care industry.

He currently works as Research Associate Director and Fellow in the Translational Science group at Johnson & Johnson supporting Global Essential Health. He is passionate about discovering new scientific insights focusing on understanding of skin physiology and the effects of topical skin care products.

He is leading a team of scientists charged with developing computational, *in vitro*, and clinical models and methods supporting the scientific credentials of J&J's skincare products. This research has led to an important number of global scientific "firsts" for J&J, including shifting the paradigm of infant skin maturation and understanding the mechanisms of cutaneous adverse reactions during oncology therapy.

Dr. Stamatas holds a PhD in Chemical/Biomedical Engineering and has co-authored more than 90 peer-reviewed publications and 14 patents.

Sustainable Smart Cosmetic Emulsions

Samiul Amin; Department of Chemical Engineering, Manhattan College

Sarah Speer, Constantina Drakontis, Department of Chemical Engineering, Manhattan College

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Introduction of research:

Development and design of sustainable cosmetic products is a key R&D priority across all the cosmetic and personal care companies. This is primarily due to the focus on addressing the strong consumer driver for cosmetic products that do not have a negative environmental footprint. This has led to the rapid development of new formulations where traditional ingredients such as synthetic surfactants, polymers, silicone oils etc are being replaced by more sustainable alternatives such as biosurfactants, biobased surfactants, biopolymers, natural oils [1, 2]. This replacement of traditional ingredients by more sustainable alternatives however is not trivial as re-building the performance of traditional ingredients is highly challenging. This is specifically true of oil-in-water and water-in-oil based emulsion systems which constitute a large majority of cosmetic products. The stability, texture/rheology/sensory has to be re-engineered in these sustainable formulations. This re-engineering of the formulations however does additionally present opportunities to build in new performance attributes.

This study will report on the formulation design of two novel sustainable emulsion systems-a thermo-responsive whey protein/chitosan oil-in-water emulsion system [3] and a glycolipid/silica particle water-in-oil emulsion system [4].

Methodology:

Whey Protein/Chitosan Oil-In-Water Emulsion*:*

A series of oil-in-water emulsions were prepared [3] keeping whey protein concentration at 15wt% and chitosan concentration at 0.5wt%. Oil-in water emulsions were prepared for 10wt% oil concentration. Oils that were explored were silicone oil, avocado oil and jojoba oil. Chitosan was added to the Whey Protein and the two were mixed. For the emulsions, oil was added to the mixture and the phases were blended at 10,000 rpm for 8 min using the IKA T18 Digital Ultra Turrax (Staufen, Germany).

Glycolipid/Silica Water-in-Oil Emulsions*:*

A series of water-in-oil emulsions were prepared [4] utilizing a mixture of either rhamnolipid/oil/silica particles with concentrations of rhamnolipid kept around 5.4%, water around 5.6%, oil around 86% and particles between 2-5% (with oil % adjusted down accordingly). Similar formulations were developed with Sophorolipid as well. The effect of particle size and concentration and biosurfactant concentration and type were investigated. Oils that were investigated included silicone oil, mineral oil, canola oil, squalene, jojoba oil.

The different amounts of biosurfactant solution and oil were added to glass vials followed by the silica particles. To ensure sufficient mixing and uniform distribution of the silica particles into the mixture, a small spatula was used for mixing until all silica particles were evenly incorporated in the formulation

Rheology Measurements: All Rheological Measurements were carried out utilizing either cone and plate or parallel plate geometry on a TA Instruments DHR rheometer

Results

Whey Protein/Chitosan Oil-In-Water Emulsion*:* One of the key objectives for this system was to utilize the thermo-responsiveness of the whey protein to design a sustainable oil-in-water emulsion system that undergoes a temperature induced rheological change. This change would potentially have both sensorial and functional benefits for make-up products such as liquid foundations. Figure 1a [3] below highlights the thermo-responsive behavior of the formulated water-in-oil emulsion system for a range of different oils. The results highlight the significant impact the type of oil utilized has in impacting the overall rheology and thermo-responsiveness.

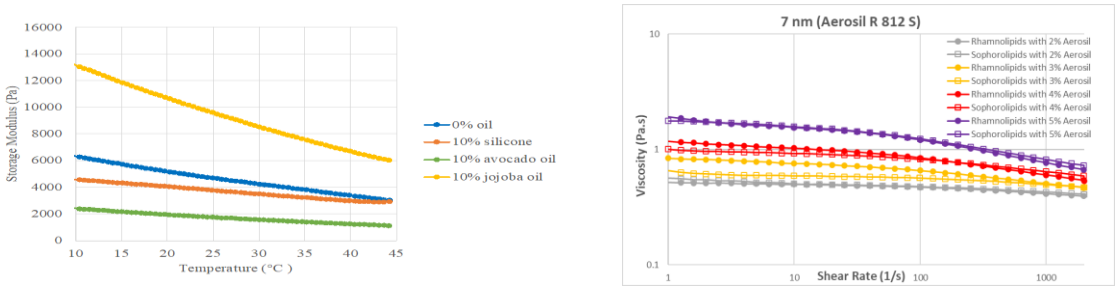

Figure 1: (a) Storage modulus change with temperature with different oils for the Whey Protein/Chitosan water-in-oil emulsion and (b) Flow Curves highlighting effect of silica particle concentration for different glycolipids for the glycolipid/silica particle water-in-oil emulsion

Glycolipid/Silica Particle Oil-In-Water Emulsion*:* One of the key objectives for this system was to design a stable water-in-oil system which has good rheological behavior. It was observed that the silica particles were structuring in the oil phase leading to a rheology build (viscosity and non-newtonian behavior) and therefore silica particle size and concentration had a big impact on the rheology. The glycolipids (Rhamnolipid and Sophorolipid) were acting as emulsifiers for the oil and not contributing to the rheology. The flow curve behavior highlighting the silica particle and glycolipid effect is illustrated in figure 1b.

Conclusion

This study highlights that that effective formulation design allows the engineering of stable oil-in-water and water in oil emulsion systems from sustainable ingredients wh

ere the rheology can be tuned to have acceptable non-netwonian character and additionally (for whey protein/chitosan) have built in smart (stimuli responsive) effects.

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About the speaker

Samiul Amin is an Associate Professor in Chemical Engineering at Manhattan College, where he leads the Cosmetic Engineering concentration area. He has over 21 years industrial and academic experience in R&D, innovation management, product design and development. His technical expertise is in formulation design of cosmetic and consumer products, colloids and complex fluids, rheology, tribology and advanced characterization. He did his PhD from NC State, MS from Johns Hopkins and BS from Rutgers University all in Chemical Engineering.