

Communication

Deodorant Efficacy of Xylityl Sesquicaprylate Vehiculated into Roll-on and Stick Prototype Formulations

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Abstract: Given the burgeoning global market for deodorant products, it is paramount to develop novel, safe, and efficacious molecules that align with the cosmetic industry's trend toward active compounds sourced from natural, sustainable, and renewable sources. In this context, we in vitro and in vivo investigated the deodorant potential of xylityl sesquicaprylate, a compound that, besides other functions, has antimicrobial activity. We performed the time–kill test to challenge the xylityl sesquicaprylate against *Staphylococcus epidermidis*, *Staphylococcus aureus*, and *Corynebacterium xerosis* and in vivo trial efficacy was established through a sniff test using two deodorant prototype formulations containing xylityl sesquicaprylate at 0.35% (*w/w*). The xylityl sesquicaprylate at 0.35% (*w/w*) in glycerin presented in vitro deodorant activity through a bactericide/bacteriostatic profile against *S. epidermidis*, *S. aureus*, and *C. xerosis*. The in vivo efficacy trial performed by the sniffers through a single application of the roll-on and the stick prototype formulations added to the developed active compound deodorant's effectiveness with a reduction in axillary bad odor, in comparison to the respective blank sample, for 2, 4, 8, and 12 h. When the deodorant efficacy was evaluated subjectively by the participants, there was always no difference between the stick sample and the blank; however, the roll-on deodorant was perceived as effective after 4 and 8 h of a single application of the sample, as established by the volunteers.

Keywords: deodorant; sniff test; time–kill kinetic test; xylityl sesquicaprylate



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1. Introduction

Body odors are among the main representatives of chemosensory communication and are related to a variety of social behaviors, which form the basis for human survival, including mate choice. It is also through body odor that newborns recognize their mothers and establish an emotional bond, motivating parental care and protection, which significantly increases child survival. In stressful situations, body odors can even be altered, inducing fight-or-flight responses in people around them, being a necessary mechanism for adapting to environmental damage and stabilizing social groups [1]. It is concluded, therefore, that body odors are a significant—if not the most significant—form of social communication between human beings [2]. Sweating is produced by sweat glands, which can be eccrine and/or apocrine and vary in density and size depending on ethnicity, gender, and body location [3,4].

Eccrine glands are distributed throughout the body, particularly on the palms of the hands and soles of the feet, being functional since birth. Its secretion, initially odorless, consists of 99% water, amino acids, ions, lactic acid, glycerol, urea, peptides, and proteins (particularly containing cysteine) [4], *Cutibacterium* (formerly *Propionibacterium*), *Staphylococcus*, and *Corynebacterium*, which are present in the skin microbiota, catabolize glycerol and

lactic acid into short-chain volatile fatty acids (C2–C3) such as acetic and propionic acids, and whose characteristic odor is found in vinegar and Swiss cheese, for example. These bacteria also degrade and transform amino acids into volatile short-chain fatty acids (C4–C5) with methyl branching, such as isovaleric acid, a characteristic odor of foot odor [5,6].

The apocrine glands only become functional after puberty. Present in greater quantities in the axilla, the secretion of the apocrine glands consists of proteins, lipids, sulfur-containing amino acids, short-chain volatile fatty acids, and steroids, such as dehydroepiandrosterone (DHEA), DHEA sulfates (DHEAS), androsterone, and testosterone [4], which contribute directly to the bad odor. Furthermore, microorganisms specific to this region, such as those of the genus *Micrococcaceae*, *Corynebacterium*, *Cutibacterium* (formerly *Propionibacterium*), and *Pityrosporum*, transform apocrine secretions into odorous substances such as (E)-3-methyl-2-hexenoic acid (3M2H—characteristic of goat odor), 3-hydroxy-3-methyl-hexanoic acid (HMHA—characteristic of cumin odor), 3-sulfanylalkanol (particularly 3-methyl-3-sulfanylhexanol; 3M3SH—characteristic and pungent odor of axillae sweat), androstenone (characteristic of urine odor) and androstenol (characteristic of musk odor) [7,8].

Added to this is the secretion of the sebaceous glands. Initially odorless and composed of wax and cholesterol esters, cholesterol and other sterols, squalenes, hydrocarbons, the sebaceous secretion also has triglycerides in its composition, which will be metabolized by bacterial lipase present in the skin microbiota and will produce glycerol, which, in turn, generates short-chain volatile fatty acids such as isobutyric acid (characteristic rancid odor) [4,9], among others.

The deodorant market globally reached USD 27.4 billion in 2022, being expected in 2023–2028 to attain a growth rate (CAGR) of 4.6% [10]. The USA leads the market for this type of product, followed by Brazil [11,12]. Among the main deodorant ingredients on the market, farnesol and triclosan stand out. Despite still being present in more than 8% of deodorants launched worldwide, studies suggested that triclosan may be related to endocrine dysfunction, neurotoxicity and cancer [13–15]. In fact, due to these characteristics, its use in cosmetic products was banned and/or restricted around the world [16].

The representation of farnesol in this category was presented, in 2022, in Germany, France, United Kingdom, and USA, where triclosan had already been replaced, reaching the point of being in 16.78% of the products launched [17]. Moreover, studies demonstrated that farnesol could be associated with allergic contact dermatitis [18,19], and restrictions on its use in deodorant products could be applied in the future. It is then necessary to develop and present to the market new safe and effective molecules that can act as deodorant agents and are aligned with trends in the cosmetic segment that constantly seek substances of natural, sustainable, and renewable origins. In this way, we in vitro and in vivo investigated the deodorant potential of xylityl sesquicaprylate, a compound that, besides other functions/attributes, has antimicrobial activity and, therefore, can act as a new deodorant ingredient for formulations in this category.

Several strategies are commonly used to control bad odor in the cosmetic segment [4,20]. Among them are odor-neutralizing agents, such as cyclodextrins and silicates that absorb moisture, reducing the growth of microorganisms and that can also absorb volatile fatty acids; enzymatic inhibitors of bacterial transformation reactions such as zinc chelators; direct inhibitors of amino acylase, the main enzyme related to the release of volatile fatty acids; and deodorant agents (antimicrobial, bactericidal, and antifungal), whose action control the growth of microorganisms, therefore reducing bad odor [4,9]. According to Nogueira et al. [16], xylityl sesquicaprylate (a mixture of mono- and diesters of caprylic acid and hexitol anhydrides derived from xylitol) is an active antiseptic ingredient derived from natural and sustainable sources, such as the by-products of cereals and vegetable oils sustainably obtained. Besides having an antimicrobial action against numerous microorganisms of interest to the pharmaceutical and cosmetic industries, xylityl sesquicaprylate was described as a solubilizer, emollient, and surfactant, since its molecule has an amphipathic characteristic. It is also noteworthy to mention that active ingredients from natural origins are a growing demand for consumers of cosmetics, globally, including deodorants. In

this scenario, bacterial and plant extracts, and essential oils are potential candidates as deodorant active ingredients [21].

In this research work, we performed the time–kill test to challenge xylityl sesquicaprylate against *Staphylococcus epidermidis*, *Staphylococcus aureus*, and *Corynebacterium xerosis* and in vivo trial efficacy was established using the sniff test for two deodorant prototype formulations containing xylityl sesquicaprylate at 0.35% (*w/w*).

2. Materials and Methods

2.1. Samples

For this investigation, xylityl sesquicaprylate was challenged as a free active ingredient for the in vitro test and was incorporated into two deodorant prototypes for the in vivo assays. Tables 1 and 2 describe the qualitative composition of the deodorants.

Table 1. Qualitative composition of the roll-on prototype deodorants.

Blank Sample (Placebo)	Xylityl Sesquicaprylate-Containing Sample
Aqua, disodium EDTA, xanthan gum, glycerin, Unibase DE (emulsifying agent), cetyl alcohol, Omega Plus (emollient), Emulfeel SGP (emulsifying agent), benzyl alcohol, caprylyl glycol, and perlite	Aqua, disodium EDTA, xanthan gum, glycerin, Unibase DE (emulsifying agent), cetyl alcohol, Omega Plus (emollient), Emulfeel SGP (emulsifying agent), benzyl alcohol, caprylyl glycol, and perlite
	Xylityl sesquicaprylate 0.35% (<i>w/w</i>)

Table 2. Qualitative composition of the stick prototype deodorants.

Blank Sample (Placebo)	Xylityl Sesquicaprylate-Containing Sample
Aqua, propanediol, glycerin, isopentyl diol, sodium stearate, perlite, benzyl alcohol, and caprylyl glycol	Aqua, propanediol, glycerin, isopentyl diol, sodium stearate, perlite, benzyl alcohol, and caprylyl glycol
	Xylityl sesquicaprylate 0.35% (<i>w/w</i>)

2.2. Time–Kill Kinetic Test

The time–kill test was performed as previously described by Mussi et al. [22] and Nogueira et al. [16]. The xylityl sesquicaprylate was tested at 0.35% diluted in glycerin against *Staphylococcus epidermidis* (ATCC 12228), *Staphylococcus aureus* (ATCC 6538), and *Corynebacterium xerosis* (ATCC 373). The microorganisms were prepared under standardized and ideal conditions. In this test, the contact times were 15, 30, 45, 60, 120, 360 (6 h), and 1440 min (24 h).

2.3. In Vivo Trial by Sniff Test and Participants' Evaluation

Only individuals who met the requirements according to the selection criteria (Table 3), and who understood, accepted, and signed the Informed Consent Form were included in the study. After the signing, participants received a copy of the consent form containing all the study information and they were sent to initial clinical evaluations to check the inclusion and non-inclusion criteria with a physician specialized in dermatology.

During 7 days (washout period), the volunteers were instructed to use a glycerin soap, in addition to not using any product on their axilla or shaving the investigational area. After this period of time, all participants were subjected to an aluminum residue detection test. In case of a positive result, the volunteer must be removed from the study due to the use of antiperspirant products during the washout period.

Efficacy assays were performed on a roll-on deodorant prototype containing xylityl sesquicaprylate 0.35% compared to its placebo, as well as a deodorant stick sample added to the same amount of the active ingredient compared to its respective blank sample (stick placebo).

Table 3. Criteria to select the participants.

Inclusion	Exclusion
Participants aged between 18 and 60 years old	Pregnancy or lactation
	Skin marks in the experimental area that interfere with the assessment of possible skin reactions
Moderate sweating	Smokers
	People with respiratory problems and participants who cannot smell properly
Intact skin in the investigational region	Hyperhidrosis
	History of adverse reactions to deodorants or antiperspirants
Agreement to follow the trial procedures and attend the clinic on the days and times determined for applications and evaluations	Other conditions considered by the evaluating physician to be reasonable for disqualification from participation of the study

After the aluminum test, participants washed their axilla with glycerin soap, followed by a baseline assessment (T0) of odor conducted by three trained sniffers with high olfactory sensitivity to assess the intensity of axillary odor, and by the participants themselves (subjective test). The intensity of the odor was assessed using a scale ranging from 0 to 10 (0: no bad odor and 10: extremely strong bad odor). The assessment took place at a distance of approximately 10 cm in a standardized way.

The research assistant applied the product randomly to the participants' axilla. Subjects were dismissed and instructed to return after 2, 4, 8, 12, and 24 h, when new odor assessments were carried out by sniffers and participants themselves along the same lines as the first assessment. During the interval between product application and evaluations, participants did not use products or wash the test area. Once the assessment was completed, participants were dismissed and the study ended (ASTM E1207-12 Standard Guide for Sensory Evaluation of Axillary Deodorancy) [6]. The experimental data were statistically treated by the *t*-Student test being the differences among the compared samples significant at the *p*-value < 0.05 (confidence interval of 95%).

3. Results and Discussion

Intense sweating or hyperhidrosis, particularly located in the axilla and soles of the feet, leads to the formation of unpleasant odors that can cause embarrassment and impact self-confidence [23]. Hyperhidrosis results from excessive sweat secretion. With an excessive amount of water in which bacteria can grow, hyperhidrosis is often accompanied by bromhidrosis, osmidrosis, or offensive body odor [4,24]. Sweat is one of the main body thermoregulation mechanisms, where its evaporation from the skin surface causes heat loss, which, consequently, reduces body temperature and protects the body's structures that have an optimal operating temperature [25]. In the case of foot odors, the maceration of the stratum corneum of the soles of the feet also contributes to an environment favorable to the growth of microorganisms, which, due to the body weight constantly supported by these structures, provides more proteins and lipids to be metabolized by the microbiota [26,27]. Furthermore, secondary infections commonly caused by fungi (mycoses) contribute to the bad odor. In fact, skin mycoses affect around 25% of the world's population. Among the most common foot mycoses are athlete's foot (*Tinea pedis*) and nail mycosis (*Tinea unguium*) whose dominant fungal species are *Trichophyton rubrum* and *Trichophyton mentagrophytes* var. *interdigitale* [28].

The time–kill kinetic test, also known as “suspension test or suspension time elimination analysis”, determines the time required for a given concentration of antimicrobial agent to kill a microorganism. Antimicrobial activity is evaluated quantitatively, showing the

effective reduction of the microbial population as a function of contact time [29]. Essentially the potential antimicrobial agent is brought into contact with the microorganisms at a time zero and cultured so that, at specified time intervals, microbial populations are evaluated. The resulting data for the time–kill test are typically presented graphically, where colony counts (CFU/mL or log) for the antimicrobial agent are plotted against time [16,22].

The choice of the xylityl sesquicaprylate concentration at 0.35% (*w/w*) was based on its minimum inhibitory concentration (MIC) performance (data not shown) against *S. aureus* (MIC = 0.30%) and *C. xerosis* (MIC = 0.25%) also indicated by economic issues to consider its use in cosmetics/dermocosmetics. Prior knowledge of the molecule activity associated with our research group expertise suggests that more complex in vitro assays, like the time–kill test, are more assertive in guiding the best choice of this active ingredient concentration.

The xylityl sesquicaprylate 0.35% (in glycerin) responded to the time–kill assay with a 99.9% reduction of the number of initial colonies of the *S. epidermidis*, *S. aureus*, and *C. xerosis* at all the tested times (15, 30, 45, 60, 120, 360, and 1440 min). It is noteworthy to mention that even after 15 min of the microbial significant reduction, the active compound kept the growth of the microorganisms under control, gradually diminishing the count of the colonies for up to 24 h (1440 min), suggesting a relevant deodorant action. Reinforcing our results, xylityl sesquicaprylate (0.45%) was challenged by the time–kill test (time of contact of 30 and 60 s) against several microorganisms involved with dental caries and periodontal issues [16], developing greater activity against *Actinomyces viscosus*, *Fusobacterium nucleatum*, *Porphyromonas gingivalis*, and *Tannerella forsythia* that corroborated with our investigation in which we observed a bactericide/bacteriostatic profile of the xylityl sesquicaprylate.

At the beginning of the in vivo trial, we observed that right after washing the axilla (T0) with the glycerin soap, the presence of bad odor in the investigational sites was perceived by the participants, as demonstrated by the scores being compatible with this type and intensity of odor, probably due to the 7 days without using deodorants (washout period) that preceded the evaluation.

The in vivo efficacy test regarding the roll-on and the stick deodorant prototypes was concluded with 19 and 22 subjects, respectively. It is worth mentioning that both the roll-on and the stick samples were prepared without silicones, being in line with more sustainable and green market trends, which are controversial due to their environmental impact (non-biodegradability and water contamination) [30]. The roll-on sample containing the xylityl sesquicaprylate 0.35% developed a significant efficacy profile in comparison with its blank version after 2, 4, 8, and 12 h after one single application (p -value < 0.05), according to the sniffers (Figure 1). After 24 h of use, no significance was found between the active-containing sample and the blank one. From the subjects' evaluation, the odor was more pronounced on the sites treated with the blank sample after 2, 4, 8, 12, and 24 h, however, presenting distinct results after 4 and 8 h (p -value < 0.05). It is reasonable to consider such distinct profile responses between the sniffers and the participants as acceptable since the panelists developed refined skills to correlate an individual odor perception calibrated in intensity. Despite the validity of the self-evaluation protocols, it may be expected that they may be less sensitive [21].

When the stick formulation was investigated, we found differences (p -value < 0.05) between the xylityl sesquicaprylate-containing (0.35%) sample and the control one (blank) in terms of the points attributed by the sniffers 2, 4, 8, and 12 h after a single application. When the points were attributed by the subjects, we observed a tendency towards greater odor from the sites treated with the blank stick, although the p -value was superior by 0.05 for all the times investigated (Figure 2).

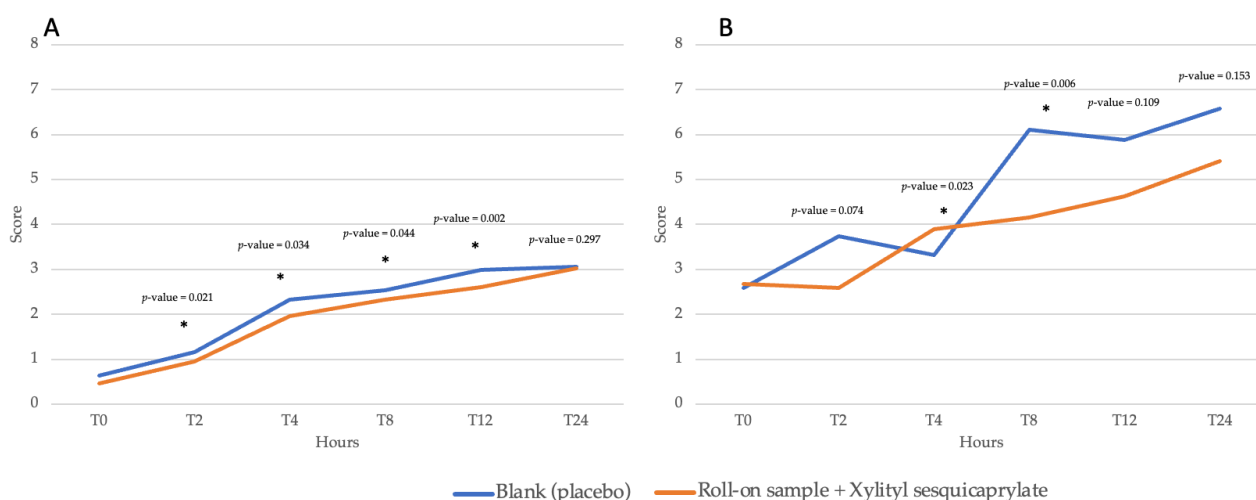


Figure 1. Scores from the sniffers (A) and the participants (subjective test) (B) for the roll-on sample. * = p -value < 0.05.

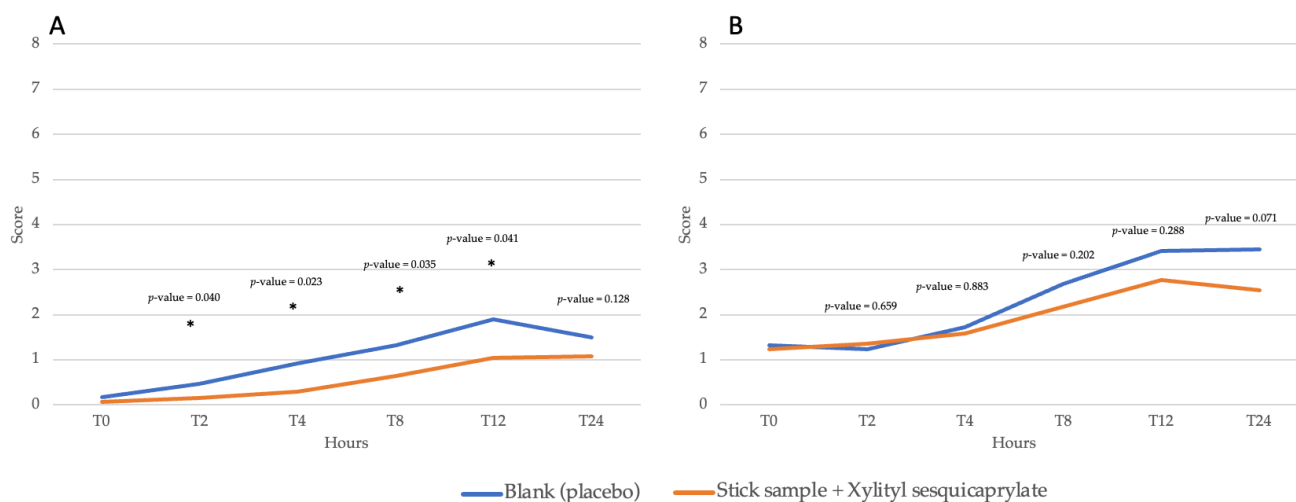


Figure 2. Scores from the sniffers (A) and the participants (subjective test) (B) for the stick sample. * = p -value < 0.05.

Several strategies are commonly used to control bad odor in the cosmetic segment [4,20]. Among them are odor-neutralizing agents, such as cyclodextrins and silicates that absorb moisture, reducing the growth of microorganisms and that can also absorb volatile fatty acids; enzymatic inhibitors of bacterial transformation reactions such as zinc chelators; direct inhibitors of amino acylase, the main enzyme related to the release of volatile fatty acids; and deodorant agents (antimicrobial, bactericidal, and antifungal), whose action control the growth of microorganisms, therefore reducing bad odor [4,9]. According to Nogueira et al. [16], xylityl sesquicaprylate is an active antiseptic ingredient derived from natural and sustainable sources, like by-products of cereals and vegetable oils sustainably obtained. Besides having an antimicrobial action against numerous microorganisms of interest to the pharmaceutical and cosmetic industries, xylityl sesquicaprylate was described as a solubilizer, emollient, and surfactant, since its molecule has an amphipathic characteristic.

4. Conclusions

Xylityl sesquicaprylate at 0.35% (w/w) in glycerin presented in vitro deodorant activity with a bactericide/bacteriostatic profile against *S. epidermidis*, *S. aureus*, and *C. xerosis*. The in vivo efficacy trial, performed using sniffers and applying a single application of roll-on and stick prototype formulations containing xylityl sesquicaprylate at 0.35% (w/w) devel-

oped deodorant effectiveness with a reduction of axillary bad odor in comparison to the respective blank sample (placebo), at 2, 4, 8, and 12 h after application. When the deodorant efficacy was evaluated subjectively by the participants, there was always no difference between the stick sample and the blank; however, the roll-on deodorant was perceived as effective at 4 and 8 h after a single application of the sample, as established by the volunteers. As a perspective aligned with our results, we may consider xylityl sesquicaprylate to be a robust alternative to the development of deodorants for a variety of formulations (like in the form of a roll-on and a stick) in comparison to the traditional/classic agents. In addition to its deodorant action *in vivo*, its origin from sustainable sources is a supplementary favorable property for innovative formulas.

5. Patents

We confirm the following patent: Xylitol esters and ethers applied as alternative emulsifiers, solvents, co-emulsifiers, and preservative systems for pharmaceutical and cosmetic products; US 8,716,506 B2; 2014.

Author Contributions: Conceptualization, L.M. and W.V.M.; methodology, L.M., C.N. and F.B.d.C.J.; formal analysis, L.M., C.N., F.B.d.C.J., A.R.B. and W.V.M.; investigation, L.M. and W.V.M.; writing—original draft preparation, L.M., A.R.B. and W.V.M.; writing—review and editing, L.M., A.R.B. and W.V.M.; supervision, W.V.M.; project administration, W.V.M.; funding acquisition, A.R.B. and W.V.M. All authors have read and agreed to the published version of the manuscript.

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Institutional Review Board Statement: This study was conducted according to the guidelines of the Declaration of Helsinki and approved by the Ethics Committee from São Francisco University, Brazil (protocol 6.646.347; CAAE 76209423.2.0000.5514; date of approval 9 February 2024).

Informed Consent Statement: Informed consent was obtained from all subjects involved in the study.

Data Availability Statement: The data presented in this study are available upon request from the corresponding authors.

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Conflicts of Interest: Authors L.M., C.N., F.B.d.C.J., and W.V.M. were employed by Chemyunion Ltd. L.M. and C.N. are co-inventors of the patent “Xylitol esters and ethers applied as alternative emulsifiers, solvents, co-emulsifiers, and preservative systems for pharmaceutical and cosmetic products” (US 8,716,506 B2). The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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