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"Investigating the Root Cause of an Unwanted Odor in Lotion: A Case Study of Metabisulfite Decomposition"

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1. Introduction

Maintaining the sensory appeal of cosmetic products is paramount for consumer satisfaction and brand reputation. Unpleasant odors can significantly detract from the consumer experience, leading to product returns, negative reviews, and damage to brand image. While fragrances are often key to consumer loyalty, preservatives are crucial for extending product shelf life, and can, sometimes, be sources of sensory deviations. Preservative degradation is a frequently overlooked yet crucial aspect that can contribute to off-odors in cosmetic formulations. This study focuses on the investigation of an unusual garlic and onion-like odor reported by consumers using a commercially available lotion. The aim was to identify the root cause of this odor and propose preventative measures to ensure future product quality. The investigation is particularly relevant considering the impact of skin pH on product stability and sensory perception. The body's skin pH typically ranges from 4.0 to 7.0, varying by location (Farage et al., 2018). This acidic pH, often referred to as the "acid mantle," is crucial for maintaining the skin's physicochemical properties and protective functions. Factors such as age, race, gender, body site, biochemical differences, and even washing habits influence the pH of the stratum corneum, thus impacting the potential for interactions between cosmetic formulations and the skin. This case study emphasizes the importance of comprehensive investigations into odor complaints, highlighting the critical interplay between formulation, preservative stability, and individual consumer factors.

2. Materials and Methods

2.1 Consumer Complaint Data Acquisition: Reports of an unpleasant garlic- or onion-like odor were compiled from online consumer feedback platforms and the company's internal cosmetovigilance system. Data collected included batch number, frequency of product use, and relevant individual characteristics where available.

2.2 Batch Analysis: Multiple batches of the implicated lotion were obtained. An olfactory analysis was performed by trained fragrance evaluators in order to characterize which batches reproduced the odor described by consumers.

2.3 Initial Chromatographic Analysis: Analysis of volatile organic compounds by headspace gas chromatography with mass spectrometry (GC-HS-MS) was performed on lotions from batches with consumer complaints about the presence of an unpleasant garlic odor and from batches without complaints. 2 g of lotion were analyzed for volatile organic compounds using headspace gas chromatography (GC-HS) coupled to mass spectrometry (MS) employing a single quadrupole (Agilent-7890B) with splitless injection mode on a HP-5 MS column, 30 m, 0.25 mm, 0.25 μm film thickness (J&W Ultra Inert Column), with the following oven program: 50 °C (3 min), heating rate of 10 °C min-1 to 300 °C (3 min), and injector temperature of 280 °C. Helium was used as carrier gas (1.0 mL. min-1). The mass spectrometer was operated in positive electron mode at 70 eV, with a range of 50 to 650 Da. The program used for the analysis of the GC-HS-MS data was MassHunter (version B.07.001413). The analytes were evaluated and identified by comparing the mass spectra and time index with the NIST reference library. The analysis focused on compounds commonly associated with garlic and onion aromas.

2.4 Replication of Consumer Usage Conditions: To confirm odor reproducibility, consumer application procedures were meticulously investigated. Reports were gathered from store managers (directly interacting with consumers) and the cosmetovigilance team (collecting feedback from official platforms). Online consumer reviews from third-party complaint platforms were also reviewed. Specific application characteristics potentially contributing to odor generation were identified. Only a subset of consumers reported the unpleasant odor, describing it as resembling spices, garlic, or onion. Some comments indicated post-shower application, suggesting that moisture, humidity and individual factors such as skin pH variations could be influential.

2.5 Ingredient Isolation and Analysis: Given inconclusive results from initial chromatographic analysis, a strategy of isolating and analyzing individual ingredients from the lotion's base formulation was implemented. Active ingredients, preservatives and fragrance exclusively used in this variation of the line have been isolated. To determine the causative agent, each component (active ingredients, preservatives, and fragrance) was individually added to portions of the base formulation, both in isolation and in various combinations (e.g., preservative and fragrance; preservative and active ingredient; fragrance and active ingredient), investigating also the interaction between these components. Descriptive sensory analysis evaluated the olfactory profile of each portion. The odor was reproduced in base formulation portions lacking both fragrance and active ingredients, indicating that the odor was not correlated with the fragrance or active ingredients but rather with the preservative component.

2.6 Advanced Analytical Techniques: The descriptive sensory analysis evaluated the olfactory profile of each portion and allowed the identification of the preservative component, sodium metabisulfite ($Na_2S_2O_5$), as a possible source of the bad odor. Thus, after the initial VOC analysis by GC-HS-MS, the formulation containing sodium metabisulfite ($Na_2S_2O_5$), known to generate sulfur dioxide (possible source of the bad odor), was analyzed in isolation. This specific analysis involved the submission of sodium metabisulfite ($Na_2S_2O_5$) to GC-HS-MS at different pHs (6.5, 7 and 7.5) in aqueous medium, and the analysis as detailed in *item 2.3*.

3. Results

The initial investigation by GC-MS did not discriminate only VOCs commonly associated with garlic or onion aromas, indicating that the odorous compounds were below the detection limit or had physicochemical characteristics that prevented their effective analysis under the conditions used. To identify the odor source, a targeted investigation was undertaken involving the isolation and individual analysis of each formulation ingredient. This approach identified sodium metabisulfite ($Na_2S_2O_5$) as the origin of the unpleasant odor. Sodium metabisulfite ($Na_2S_2O_5$) hydrolysis, accelerated in acidic media, generates bisulfite ions (HSO_3^-), which decompose to release sulfur dioxide (SO_2) (Figure 1), progressively and under the action of temperature (reaction initiated at 30 °C). SO_2 is characterized by a pungent odor and potential toxicity (Atkinson et al., 1993; Ali et al., 2024). GC-HS-MS analysis confirmed the presence of SO_2 (m/z 48 and 66, Figure 2) and an unidentified amino compound. Amine-containing compounds, in the presence of sulfur dioxide, can form thiols (-SH), known for their characteristic garlic and onion odors, and are commonly found in Allium species (Block et al., 2013; Marcinkowska; Jeleń, 2022). These compounds can form and likely contribute to the observed odor profile. Significantly higher concentrations of SO_2 and amino groups were observed in samples exposed to slightly acidic aqueous conditions (pH 6.5).

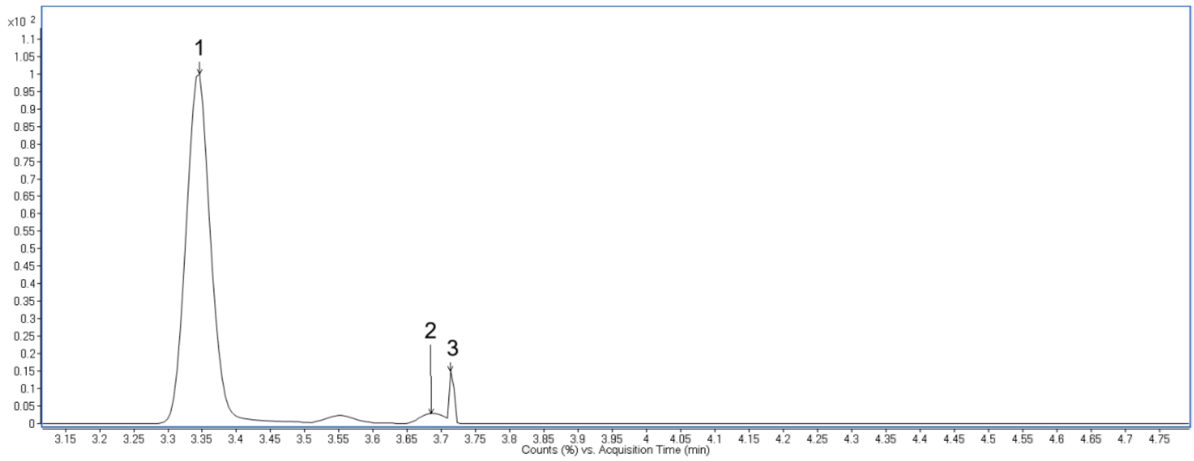

Figure 1. Total ion current (TIC) chromatogram of the base sample containing Sodium metabisulfite. 1-Sulfur dioxide; 2-Compound with amine group; 3- isopropyl alcohol from the formulation.

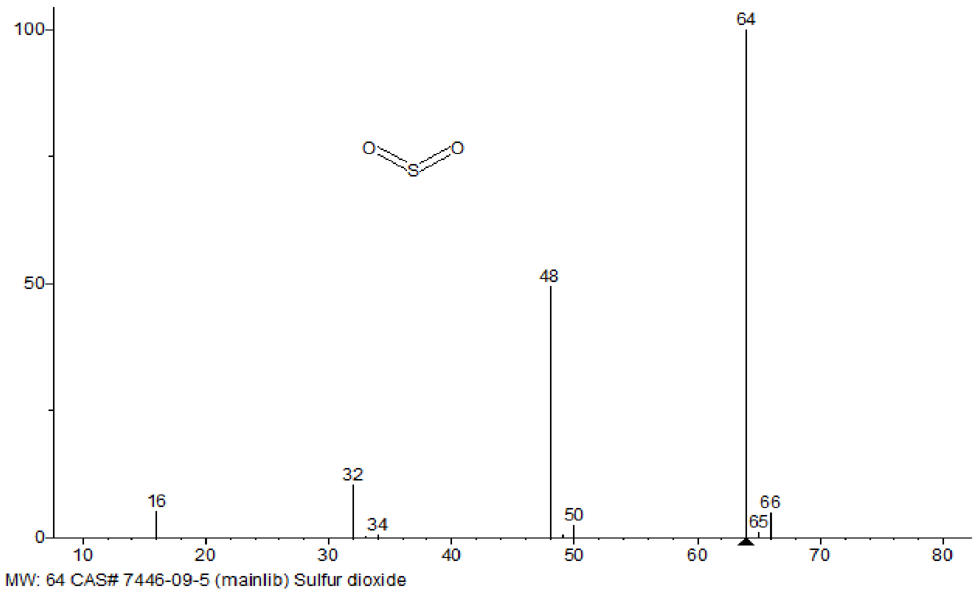

Figure 2. Mass Spectrum of Sulfur Dioxide.

4. Discussion

The results clearly demonstrate that the unpleasant garlic and onion odor present in the lotion originates from the decomposition of sodium metabisulfite ($Na_2S_2O_5$). This decomposition produces sulfur dioxide (SO_2) and biosulfite ions (HSO_3^-), which can react with amines present in the base formulation, such as triethanolamine, and thus potentially form thiols, sulfur-containing compounds responsible for the characteristic olfactory profile. The decomposition process appears to be significantly greater under catalyzed hydrolysis conditions in a slightly acidic medium. The variability in odor perception among consumers likely stems from differences in individual skin conditions, particularly pH and moisture content, especially on the hands and arms where the lotion was applied. Skin pH, typically ranging from 4.5 to 6.5 on the hands and arms, varies significantly due to factors such as genetics, hygiene practices, and environmental exposures (Lambers et al., 2019; Rawlings, 2009). A more acidic skin pH accelerates the decomposition of sodium metabisulfite, leading to increased production of SO_2 and sulfur-containing compounds. Moreover, increased moisture levels on the skin surface can also influence the rate of $Na_2S_2O_5$ decomposition and subsequent release of odorous volatiles (Draelos, 2016). The combination of lower skin pH and higher moisture levels, perhaps more prevalent in certain individuals or after showering as reported by some consumers, could create a microenvironment particularly conducive to the formation of these malodorous sulfur compounds. Therefore, individual variations in skin characteristics must be considered as contributing factors to the observed variability in odor perception.

The initial failure to detect the odor-causing compounds using standard GC-HS-MS analysis highlights the analytical challenges inherent in identifying trace volatile components responsible for subtle off-odors within complex cosmetic formulations. The low concentrations of the volatile sulfur compounds, coupled with potential matrix effects from other formulation ingredients, likely contributed to the initial inconclusive results. This necessitated a more targeted approach, employing ingredient isolation and individual analysis to determine the specific component responsible for the off-odor. This strategy proved crucial in identifying sodium metabisulfite as the source of the problem.

5. Conclusion

This case study underscores the importance of thorough investigation into consumer complaints regarding odor in cosmetic products. The identification of sodium metabisulfite as the source of the garlic/onion-like odor and the subsequent identification of SO_2, likely related to the interaction of metabisulfite degradation ions with an amino group compound present in the base formulation, as the odor-causing volatile compounds, highlights the potential for preservative decomposition to significantly impact product quality. The influence of skin pH on the decomposition rate further emphasizes the need for careful consideration of individual consumer factors during formulation. Replacing sodium metabisulfite with alternative preservatives and optimizing the lotion formulation to mitigate acidic conditions are crucial steps to prevent the recurrence of this problem. This research emphasizes the value of a multi-faceted approach to odor investigation in cosmetic product development, prioritizing consumer experience and maintaining brand integrity. Future research could explore the specific reaction mechanisms involved in the decomposition of sodium metabisulfite under different conditions and investigate alternative preservatives with enhanced stability.

6. References

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