COMBINED APPLICATION OF pH REDUCTION AND ACTIVATED CARBON FOR THE REMOVAL OF OFF-FLAVOR COMPOUNDS (GEOSMIN AND 2-MIB) IN FERMENTED CARP-BASED FISH SAUCE

Đến tòa soạn: 06-10-2025

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TÓM TẮT

ỨNG DỤNG KẾT HỢP PHƯƠNG PHÁP KHỬ pH VÀ THAN HOẠT TÍNH ĐỂ LOẠI BỎ HỢP CHẤT GÂY MÙI LẠ (GEOSMIN AND 2-MIB) TRONG NƯỚC MẮM CÁ CHÉP LÊN MEN

Các hợp chất gây mùi lạ geosmin và 2-methylisoborneol (2-MIB) thường xuất hiện trong cá nước ngọt và các sản phẩm nuôi trồng thủy sản, làm giảm chất lượng cảm quan. Nghiên cứu này đánh giá khả năng loại bỏ các hợp chất đó khỏi nước mắm làm từ cá chép bằng phương pháp kết hợp axit hóa và sử dụng than hoạt tính (PAC và GAC). Thí nghiệm Jar test theo mẻ và cột dòng chảy cho thấy việc hạ pH xuống 4 giúp tăng cường khả năng hấp phụ, trong đó PAC cho hoạt tính hấp phụ mạnh hơn, còn GAC phù hợp với xử lý liên tục ở quy mô lớn. Phương pháp kết hợp axit hóa và than hoạt tính cho thấy hiệu quả khử mùi cao hơn so với từng phương pháp riêng lẻ. Mặc dù carbon hữu cơ hòa tan và axit amin cạnh tranh vị trí hấp phụ, đánh giá cảm quan xác nhận mùi đất – mùi mốc giảm đáng kể trong khi vẫn giữ được vị umami. Kết quả chỉ ra rằng việc tích hợp axit hóa với xử lý bằng than hoạt tính là một phương pháp hiệu quả nhằm nâng cao chất lượng nước mắm từ cá nước ngọt, đồng thời mang lại lợi ích trong việc tận dụng nguồn cá chép xâm hại ở một số khu vực không có thói quen ăn cá chép.

Từ khóa*: nước mắm cá nước ngọt, geosmin, 2-MIB, than hoạt tính.*

1. INTRODUCTION

Fish sauce is an indispensable specialty in Asian meals, traditionally produced from marine seafood with a distinctive flavor. It has been exported worldwide and well-received internationally. Carp is a freshwater fish with high nutritional value and is widely farmed in Asia as an aquaculture species that provides significant income. However, in countries such as the United States and Canada, carp is not consumed despite its rapid reproduction and strong proliferation in freshwater systems. This uncontrolled growth has turned carp into an invasive species, causing serious ecological and environmental problems in freshwater ecosystems [1, 2, 3]. Some previous studies have explored the use of carp as a substitute for marine fish in fish sauce production [4, 5, 6, 7]. However, the flavor and taste of carp-based fish sauce do not fully match those produced from marine fish.

Producing fish sauce from invasive freshwater fish like carp not only creates a nutritious product but also helps control these prolific species in U.S. waters, reducing ecological damage and costly management efforts [8]. Geosmin and 2-methylisoborneol (2-MIB) are the two major off-flavor compounds commonly found in freshwater fish and aquaculture products. Although they are not considered toxic, their extremely low odor threshold concentrations (in the range of 5–10 ng/L) lead to significant sensory problems, reducing consumer acceptance and market value of aquatic-derived foods. In freshwater fish sauce produced from species such as carp, geosmin and 2-MIB have been reported at concentrations ranging from several tens to several hundreds of ng/L, which is sufficient to impart earthy and musty off-flavors [9]. Conventional treatments such as boiling or seasoning are generally ineffective due to the chemical stability and hydrophobicity of these compounds [10]. Among various control strategies, adsorption by activated carbon has been widely applied because of its high surface area and strong affinity for hydrophobic molecules [11]. However, the efficiency of activated carbon can be compromised by competition with other organic matter, leading to premature saturation of adsorption sites [12, 13]. Recent studies have suggested that adjusting pH conditions can influence the solubility, ionization behavior, and interaction of geosmin and 2-MIB with carbon surfaces, potentially enhancing removal performance [14, 15, 16]. This study investigates a combined approach using activated carbon adsorption and pH reduction to improve the elimination of geosmin and 2-MIB, aiming to provide a practical and scalable solution for improving the sensory quality of freshwater fish sauce and related products.

2. MATERIALS AND METHODS

2.1. Materials

Freshwater fish sauce was prepared from carp (Cyprinus carpio) following a traditional 3-month fermentation process with 20% (w/w) salt at ambient temperature. The resulting liquid was filtered to remove solids before use. Food-grade acetic acid, citric acid, and lactic acid were used for pH adjustment. Powdered activated carbon (PAC, 20–50 μm, micropore-rich) and granular activated carbon (GAC, 4–8 mesh, micropore-dominant) were applied as adsorbents. After PAC or GAC treatment, fish sauce was recovered by filtration and decantation. Volume loss was below 2%, and no significant changes in color, °Brix, or total nitrogen were observed, indicating that the main constituents of fish sauce were preserved. Analytical standards of geosmin and 2-MIB were used for calibration. All reagents were of analytical grade, and ultrapure water (18.2 MΩ·cm) was used throughout. PAC and GAC were dried at 105°C for 2 h before use. Fish sauce samples were stored at 4°C and analyzed within 48 h. Geosmin and 2-MIB were quantified by Solid Phase Microextraction with Gas Chromatography-Mass Spectrometry (SPME-GC-MS), amino acids were determined by High-performance liquid chromatography (HPLC), and Dissolved Organic Carbon (DOC), Total Organic Carbon (TOC), Total Suspended Solids (TSS), and pH were measured using standard methods.

2.2. Experimental design and Analytical methods

The removal of geosmin and 2-MIB from carp-based fish sauce was evaluated using

Jar test batch (Digital Jar Test Apparatus 4-Spindle) experiments and column tests. In Jar tests, 1000 mL fish sauce samples were adjusted to pH 4–6 with food-grade acids and treated with PAC at 10–100 mg/L.

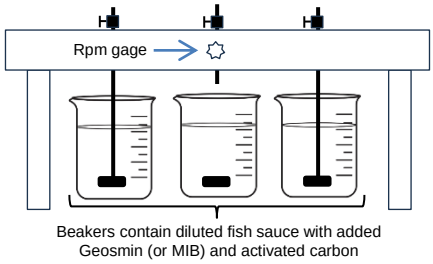

Figure 1. *Jar-test used in the study*

Samples were stirred at 150 rpm for 15–120 min to simulate mixing conditions, after which geosmin and 2-MIB were analyzed by GCMS-QP2010, Shimadzu. Column experiments were performed using a 30 cm GAC (4–8 mesh) column, with fish sauce with or without prior pH adjustment passed through at empty bed contact times of 2–10 min. The retention time was 14.75 min for geosmin and 10.72 min for 2-MIB. Effluents were collected for geosmin, 2-MIB, DOC, and amino acid analysis.

All tests were done in triplicate with blank controls, and differences between treatments were compared based on mean values and standard deviations.

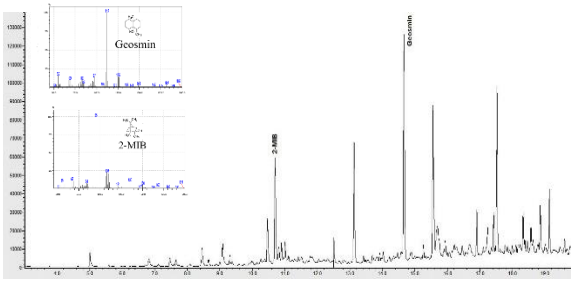

Figure 2. *Chromatogram of geosmin and 2-MIB (the overlaid inset mass spectra) in fish sauce*

3. RESULTS

3.1. Effect of acidification on Geosmin/2-MIB

Acidification of carp-based fish sauce significantly influenced the removal of geosmin and 2-MIB. Lowering the pH increased the reduction efficiency of these off-flavor compounds, and longer contact times further enhanced removal. At pH 4, geosmin concentration decreased by 45% after 15 minutes and up to 75% after 120 minutes, while 2-MIB decreased by 40% to 70% over the same period. At higher pH values (5–6), removal efficiencies were lower, indicating that acidification facilitates partial degradation or structural modification of geosmin and 2-MIB, making them more amenable to subsequent adsorption or volatilization.

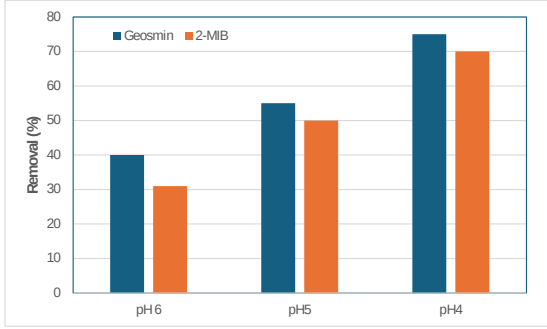

Figure 3. *Removal efficiency of Geosmin and 2-MIB under different acidification conditions*

Previous studies have also demonstrated that lowering pH can reduce the levels of geosmin and 2-MIB in aqueous systems, although the extent of reduction depends on acid type, concentration, and contact time [5]. These findings suggest that acidification alone can moderately reduce off-flavor compounds and may serve as a useful pretreatment step before applying activated carbon adsorption in the production of freshwater fish sauce.

3.2. Effect of activated carbon under different pH conditions

Activated carbon significantly enhanced the removal of geosmin and 2-MIB from carp-based fish sauce, with performance varying by carbon type, dosage, and

solution pH. In batch Jar tests, PAC achieved rapid adsorption, reaching 85% geosmin and 80% 2-MIB removal at 100 mg/L within 60 minutes, while GAC required higher doses or longer contact times for comparable performance. At neutral pH (6), removal efficiencies were moderate (40–60%), whereas acidification to pH 4 markedly improved adsorption capacity, resulting in 10–20% higher removal across all tested dosages. Breakthrough curves from column experiments confirmed this trend: at pH 4 and EBCT = 10 min, breakthrough was delayed by ~40% relative to neutral conditions, extending effective bed life.

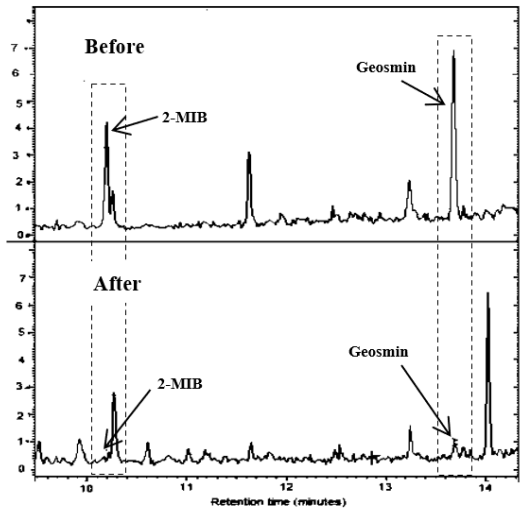

Figure 4. *Chromatogram of Geosmin and 2-MIB before (upper) and after (lower) absorption by PAC*

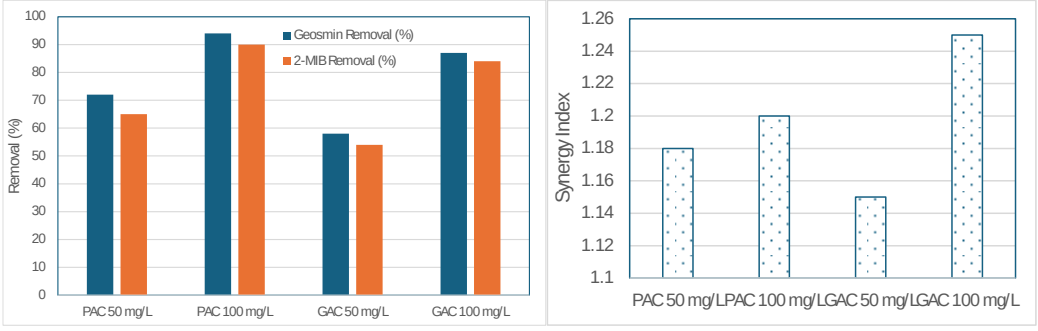

Figure 5. *Removal efficiency of geosmin and 2-MIB by PAC and GAC under acidified conditions (pH = 4) (left) Different PAC and GAC doses, (right) Synergy index at varying doses*

To quantify this enhancement, a synergy index (SI) was calculated as the ratio of combined removal efficiency (pH reduction + AC) to the sum of individual effects. SI values ranged from 1.15–1.25, indicating a synergistic effect rather than simple additivity. This synergy is likely due to changes in molecular interactions at lower pH, which increase the affinity of geosmin and 2-MIB for activated carbon surfaces. Overall, PAC is advantageous for rapid batch treatment, while GAC under acidic conditions offers a practical and scalable strategy for continuous operation.

3.3. Factors influencing the removal of geosmin and 2-MIB

The efficiency of geosmin and 2-MIB removal was not only affected by pH and activated carbon dose, but also by the matrix composition of the carp-based fish sauce. DOC and TOC acted as competitive adsorbates, occupying active sites on PAC and GAC, thereby reducing the adsorption efficiency of off-flavor compounds. At higher DOC levels (>30 mg/L), the removal efficiency of geosmin and 2-MIB decreased by 10–20% compared to samples with DOC <15 mg/L. Similarly, amino acids, particularly

glutamic acid, which contributes to umami taste, showed moderate competition with activated carbon, but also partially masked earthy-musty odors, as reflected in sensory evaluation scores.

Table 1. *Influence of DOC, amino acids attributes on geosmin and 2-MIB removal*

Parameter	Control (no AC, pH 6)	PAC (50 mg/L, pH 4)	GAC (EBCT 5 min, pH 4)
DOC (mg/L)	32.5 ± 1.2	18.4 ± 0.9	20.1 ± 1.0
TOC (mg/L)	45.8 ± 1.6	27.3 ± 1.1	29.5 ± 1.3
Glutamic acid (mg/L)	5.6 ± 0.4	4.8 ± 0.3	5.0 ± 0.2
Geosmin residual (ng/L)	135 ± 12	35 ± 5	42 ± 6
2-MIB residual (ng/L)	128 ± 10	38 ± 4	46 ± 5

Sensory analysis results demonstrated that acidification combined with activated carbon treatment significantly improved the overall flavor acceptability of the fish sauce. Panelists reported a notable decline in earthy and musty perception (mean score decreased from 7.2 to 3.1 on a 9-point scale), while umami intensity was maintained or slightly enhanced. This indicates that although matrix constituents like amino acids and DOC reduce adsorption efficiency, they may synergistically contribute to improved sensory outcomes by balancing flavor attributes.

3.4. Discussion

The results confirm that acidification and activated carbon act synergistically to enhance the removal of geosmin and 2-MIB from carp-based fish sauce. Lowering pH to 4 improved adsorption efficiency by promoting hydrophobic and π–π interactions between odor compounds and carbon surfaces, consistent with previous studies on aqueous systems [17, 18]. The calculated synergy index indicates cooperative effects rather than simple additive behavior, aligning with earlier reports of enhanced adsorption under acidic conditions [19].

Matrix components such as DOC and amino acids moderately interfered with adsorption, reducing removal by 10–20%, as similarly observed in water treatment applications [20]. However, amino acids, especially glutamic acid, helped maintain umami balance and masked residual earthy–musty odors, resulting in improved sensory scores.

Sensory evaluation confirmed significant flavor enhancement, with earthy–musty perception declining by more than half, while desirable umami notes were preserved. These findings demonstrate that controlled acidification combined with PAC or GAC treatment is an effective and practical method to improve the sensory quality of freshwater fish sauce. Moreover, the use of invasive carp species provides an environmentally beneficial approach that supports both flavor improvement and ecological management.

4. CONCLUSION

Acidification combined with activated carbon effectively removed geosmin and 2-MIB from carp-based fish sauce, with PAC showing faster kinetics and GAC offering scalable continuous treatment. Acidic conditions (pH 4) enhanced adsorption and synergistically improved removal efficiency, despite competition from DOC and amino acids. Sensory evaluation confirmed reduced earthy–

musty off-flavors while maintaining umami taste. Utilizing invasive freshwater species such as carp for fish sauce production provides both ecological benefits and a practical strategy to improve product quality.

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