



## Research article

## Activated carbon/titanium dioxide composite to adsorb volatile organic compounds associated with human body odor

Takeshi Hara<sup>\*</sup>, Haruna Nabei, Ayumi Kyuka

Fundamental Research Institute, Mandom Corp., 5-12, Juniken-cho, Chuo-ku, Osaka, Japan

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## ABSTRACT

The human body generates various malodor compounds in different body parts and even in the same parts. A more effective and versatile deodorant material that can suppress various key contributors to the body odor must be developed to improve the quality of life of affected individuals. In this study, activated carbon (AC) was found to have higher adsorption ability toward key body odor-generating compounds than zinc oxide (ZnO), which is a well-known deodorant material. To prevent pigmentation of human skin induced by the direct application of AC, white activated carbon (WAC) was developed by blending AC, titanium dioxide (TiO<sub>2</sub>), and ammonium acrylate copolymer. AC to TiO<sub>2</sub> ratio of 1:6 was found to be the optimum blending ratio to form WAC. Compared with ZnO, this optimal WAC exhibited higher adsorption ability toward five key body odor compounds. The *in vivo* evaluation of the sample containing WAC revealed that it significantly suppressed the generation of axillary odor without a bactericidal effect. The developed WAC can effectively suppress the human body odor in different body parts. These findings are valuable for individuals experiencing psychological stress attributed to their unpleasant body odor.

## 1. Introduction

Hundreds of volatile organic compounds (VOCs) are emitted from the human body. Some of these VOCs are responsible for body odor. In the modern society, body odor is a universal problem, irrespective of age or gender, because it can impart negative impressions. Body odor is mainly generated by bacterial metabolism and oxidation of sweat and lipids on the human skin. It consists of various volatile compounds such as short-chain fatty acids, ketones, alcohols, and aldehydes [1, 2, 3]. Recently, odorants are used as disease biomarkers to develop some non-invasively tools for diseases monitoring [4, 5, 6]. In a previous study, the authors identified diacetyl as a key malodor-generating compound released by the scalp, axillae, and feet [3, 7]. On the other hand, Zeng et al. discovered that in addition to acetic acid, 3-methyl-2-hexenoic acid (3M2H) is a key contributor to axillary odor [8]. Moreover, isovaleric acid and 2-nonenal were identified as odor-causing compounds on human feet and back, respectively [9, 10]. Therefore, odor-causing compounds vary on different body parts and on the same body parts of different individuals. The authors have previously reported that the axillary odor of Japanese men can be categorized into three major types: milk-like odor (type M;

57%) cumin-like odor (type C; 18%), and acid-like odor (type A; 16%) [3].

To control human body odor, antiperspirant, bactericidal, and deodorant-based techniques are commonly used [11, 12, 13]. Antiperspirants can block human sweat, but it cannot suppress the odor generation. Bactericidal agents fail in cases of malodor generation that does not involve bacterial metabolism. On the other hand, adsorbents are particularly effective in preventing body odor arising from VOCs, especially ZnO, which is frequently used for this purpose [14]. However, ZnO, which uses an ionic bonding mechanism, is only fully effective with negatively charged ionic compounds, such as short-chain fatty acids, but is insufficient for adsorption of uncharged compounds. Therefore, it is necessary to develop an effective and versatile deodorant material that can control malodor-generating compounds with different chemical structures emitted from different body parts. This product would improve the quality of life for consumers who are affected by their own body odor.

Activated carbons (ACs) are often used as excellent and versatile adsorbents. The porous surface of ACs can inactivate various chemical substances by adherence of an extremely thin layer of the compounds to the large surface area of the carbon. They are typically used for adsorptive removal of color, odor, taste, and undesirable organic and

<sup>\*</sup> Corresponding author.

E-mail address: [takeshi.hara@mandom.com](mailto:takeshi.hara@mandom.com) (T. Hara).

inorganic pollutants from drinking and industrial waters [15, 16, 17]. In addition, ACs are frequently used for the purification of many chemical, food, and pharmaceutical products [16, 18, 19, 20]. However, to the best of the authors' knowledge, no studies have investigated using ACs in leave-on cosmetic products such as deodorants.

Therefore, based on our discovery that AC exhibited high adsorption ability for various body odor-generating compounds, a new deodorant material (white activated carbon, WAC) that has an acceptable appearance on the skin was developed.

## 2. Materials and methods

### 2.1. Generals

Diacetyl, acetic acid, (2E)-3M2H (BOC sciences, NY, USA), 2-nonenal, and isovaleric acid were prepared as representatives of body odor compounds present in the scalp, axillae, back, and feet. AC and TiO<sub>2</sub> were obtained from Kuraray Co. Ltd. (Tokyo, Japan) and Ishihara Sangyo Kaisha Ltd. (Osaka, Japan), respectively. An ammonium acrylates copolymer solution (YODOSOL GH800F; Akzo Nobel N.V.) was used in the formation of WAC. ZnO with a mean diameter of 0.55 μm (Japanese Pharmacopoeia grade) was used as the control in the present study. Carboxen/polydimethylsiloxane (CAR/PDMS, an 85-μm film) fibers obtained by solid phase microextraction (SPME), polypropylene hold caps, and polytetrafluoroethylene/silicone septa were purchased from Supelco Inc. (Belfonte, PA, USA). All the other chemicals were purchased from FUJIFILM Wako Pure Chemical Corporation (Osaka, Japan). All experimental protocols were approved by the Human Ethics Committee at Mandom Corp. The purpose and procedures of the studies were explained to all subjects before their informed consent was obtained.

### 2.2. Adsorption tests of AC and ZnO

Propylene glycol was used to prepare diacetyl (0.1% (v/v)), 2-nonenal (0.1% (v/v)), acetic acid (0.5% (v/v)), 3M2H (1.0% (v/v)), and isovaleric acid (0.5% (v/v)). First, an aliquot (20 μL) of each sample solution and AC (30 mg) or ZnO (30 mg) was applied on cotton pads (1.5 cm × 1.5 cm). Each pad was transferred into sealed amber vials (40 mL). An SPME fiber was inserted into each vial through the seal septa. Next, each vial was heated to 37 °C for 30 min to remove the volatile compounds generated by the cotton.

### 2.3. Development of WAC

AC powders (1 g) with a mean diameter of 9.4 or 40.5 μm were fully mixed with ammonium acrylates copolymer solution (0.9% (v/v), 1.5 g) in ultrapure water. With a mean diameter of 0.25 μm, TiO<sub>2</sub> powders in varying concentrations of 5–20 g were added to the mixture to fabricate corresponding WACs. After drying the samples for 2 h at 115 °C, WAC was obtained by uniform grinding using a crusher (Figure 1). The L\* value (whiteness) of the WAC samples was measured using a spectrophotometer (CM-2600d, Konica Minolta, Japan). The lightnesses of the samples were then determined on a scale ranging from black (0) to white (100). WAC, AC, and ZnO (10 mg each) were spread by a spatula on the inner forearm of the human subjects. The color of the samples was then visually evaluated on human skin. The adsorption test of WAC was conducted using each odor compound (20 μL) and WAC (30 mg) as described above.

The adsorption abilities of pristine AC were compared to those of AC contained in the WAC. For this purpose, equivalent amounts of AC and WAC (4.3 and 30 mg; equivalent to 4.3 mg of AC, respectively) were used. The adsorption levels of all the WAC samples were quantified using solid phase microextraction followed by gas chromatography–mass spectrometry (SPME–GC–MS).

### 2.4. In vivo WAC deodorant test

A spray sample, including 1.5% (w/w) WAC, 3.5% (w/w) isopropyl myristate, and 95% (w/w) liquefied petroleum gas, was used to test the material *in vivo*. A total of eight subjects washed their axillae with non-perfumed soap and applied the sample on one axilla. The odor intensities of both axillae were evaluated after 6 h by five assessors. The odor intensity was scored as follows: 0 = none; 0.5 = between 0 and 1; 1 = very faint; 1.5 = between 1 and 2; 2 = faint; 2.5 = between 2 and 3; 3 = noticeable; 3.5 = between 3 and 4; 4 = strong; 4.5 = between 4 and 5; 5 = very strong. The increase rate of each axillary odor was calculated as the difference between the odor intensities at the start time of the test and at 6 h of testing. Here, the odor intensity was considered as an interval scale. Simultaneously (after the 6 h), the axillary microbes were collected by rubbing the axillae (70 cm<sup>2</sup>) using sterile cotton swabs soaked in phosphate buffer saline solution. The standard curve was plotted based on *Staphylococcus epidermidis* ATCC14990 cultured in trypticase soy broth at 37 °C. The adenosine triphosphate (ATP) yield was measured based on fluorescence using a Rucifel-HS kit (Kikkoman, Japan) and an ATP

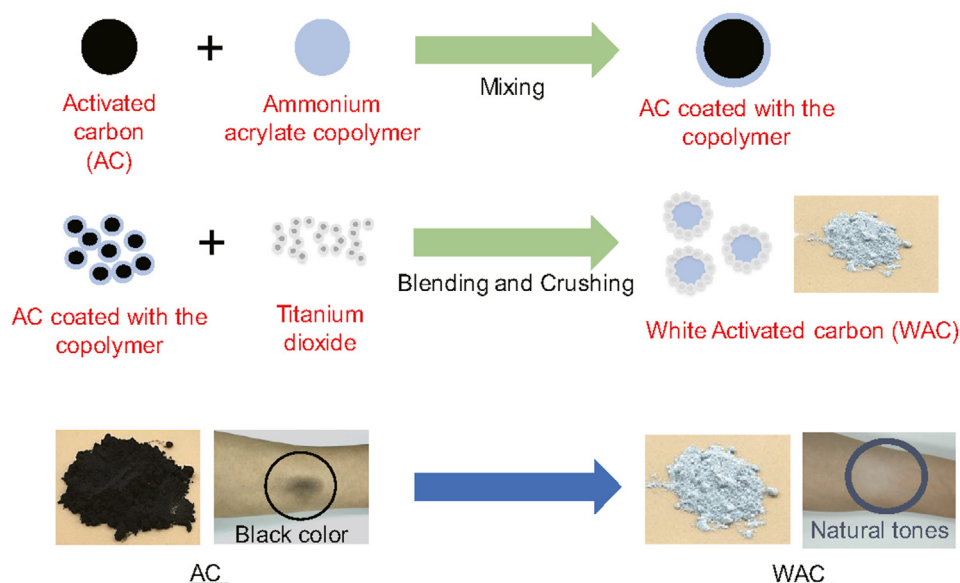


Figure 1. Schematic representation of the development of WAC.

analyzer (Lumitester C-110, Kikkoman) following the instructions of the manufacturers. Both axillary viable microbial counts were calculated from the standard curve of the intracellular ATP yield of *S. epidermidis*.

### 2.5. GC–MS analysis

The extracted compounds were desorbed from the SPME fiber in the GC–MS injector at 250 °C for 5 min. The GC–MS analysis was performed on a 7890B GC instrument equipped with a 5977A (inert XL EI/CI MSD with a triple-axis detector) mass spectrometer (Agilent Technologies, USA). A DB-free fatty acid phase capillary column (60 m × 0.25 mm with a 0.50- $\mu$ m film; J&W Scientific, USA) was used. Helium was used as the carrier gas at a constant flow rate of 2 ml/min. The volatiles were injected in a splitless mode at 220 °C. The oven temperature program started at 35 °C and remained at this temperature for 3 min. The temperature then increased to 140 °C at a rate of 7 °C/min. Finally, the rate increased to 15 °C/min until it reached 220 °C, which was maintained for 9 min. The effluent was transferred to the mass detector to measure the mass spectra by electron impact at 70 eV, a transfer temperature of 250 °C, and a source temperature of 230 °C. Chromatogram spectra were recorded and processed using the Enhanced Chemstation software for GC–MS (Agilent Technologies, USA). The fragment ions were studied for quantification at mass to charge ratios ( $m/z$ ) = 86, 60, 100, 70, and 87 for diacetyl, acetic acid, 3M2H, 2-nonenal, and isovaleric acid, respectively. The adsorption levels of the samples were calculated using the peak areas of the odor compounds determined by the GC–MS and calculated as follows:

$$\% = [(1 - \text{peak area of each odor compound in each adsorbent} / \text{peak area of each odor compound in the control})] \times 100$$

### 2.6. Statistical analysis

All statistical analyses were performed using Excel (Office 365) with Statcel4 software (OMS Publishing Inc., Saitama, Japan). Different statistical tests were used for different experiments as indicated in the description of each figure.

## 3. Results and discussion

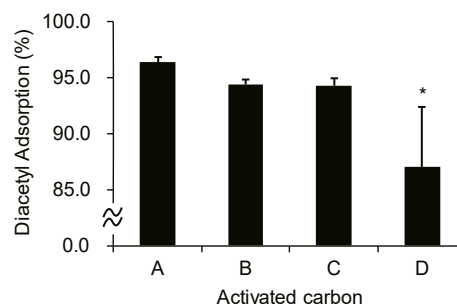
### 3.1. Evaluation of the adsorption of body odor compounds on AC

ACs are versatile adsorbents. The adsorption ability of AC toward the key malodor compounds present in the scalp, axillae, back, and feet was compared to that of ZnO as a control. AC exhibited much higher adsorption abilities toward diacetyl and 2-nonenal compared to ZnO (Figure 2). In contrast, no significant differences were observed between AC and ZnO during the adsorption of 3M2H and isovaleric acid (Figure 2), suggesting that ZnO neutralizes fatty acids, such as acetic acid,

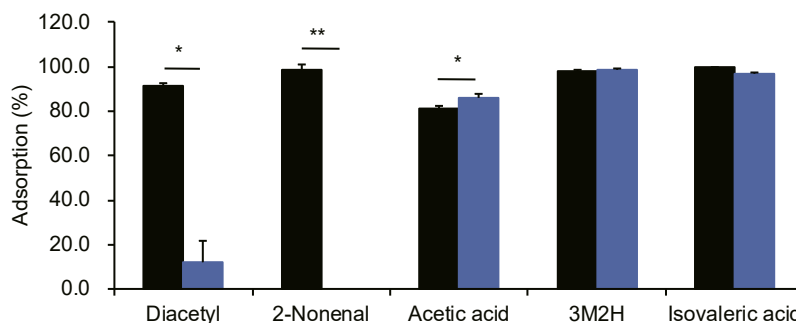
3M2H, and isovaleric acid, rendering them inactive. However, non-charged compounds, including diacetyl and 2-nonenal, were not adsorbed. The adsorption mechanism of AC involves physical adsorption and chemisorption [15, 16]. Physical adsorption involves binding odor compounds to the surface of ACs by van der Waals forces. Chemisorption, on the other hand, takes place through a chemical reaction, which involves exchange or sharing of electrons between the adsorbate molecules and the surface of the adsorbent, resulting in the formation of chemical bonds. Therefore, these two adsorption mechanisms contributed to the adsorption of body odor compounds with various functional groups. These results suggest that, compared to ZnO, AC is a more efficient material for controlling the generation of various body malodor compounds, including ionic and non-ionic compounds.

### 3.2. Screening ACs for WAC formation

The adsorption capacity of ACs greatly depends on the pore size, surface area, and apparent density [21]. To select the AC type with higher adsorption capacity to the odorant, 4 ACs were screened using acetic acid. Carbons A and D exhibited the highest and lowest adsorption abilities, respectively, toward acetic acid (Figure 3). The reason is that carbon D showed the lowest iodine adsorption, which was used to determine the surface area and apparent density of ACs, compared to the other tested carbonaceous materials (Table 1). Although carbon C has almost the same apparent density and even larger surface area (iodine adsorption) compared to carbon A, the adsorption ability was lower than that of carbon A (Figure 3, Table 1). This suggests that the bulk and surface structures as well as the pore size of ACs also affect the adsorption of the odorants and are important to effectively suppress body odor.



**Figure 3.** Adsorption abilities of various types of ACs for acetic acid (0.5% (v/v)) odorant. Data were collected in three independent experiments and are expressed as their means  $\pm$  standard deviations ( $n = 3$ ). \*:  $p < 0.05$  versus activated carbon A, one-way ANOVA followed by Dunnett's test for multiple comparisons.



**Figure 2.** Adsorption abilities of AC and ZnO toward body malodor compounds. Black and blue bars indicate AC and ZnO, respectively. Data are shown as three independent experiments and expressed as means  $\pm$  standard deviation, where  $n = 3$ . \*:  $p < 0.05$  and \*\*:  $p < 0.01$  (by Welch's t-test).

**Table 1.** Characteristics of the activated carbons subjected to screening.

Carbon	Apparent density (g/mL)	Iodine adsorption (mg/g)	Pore size (Å = 0.1 nm)
A	0.499	1120	8.47
B	0.504	1110	8.47
C	0.490	1890	13.06
D	0.403	790	8.62

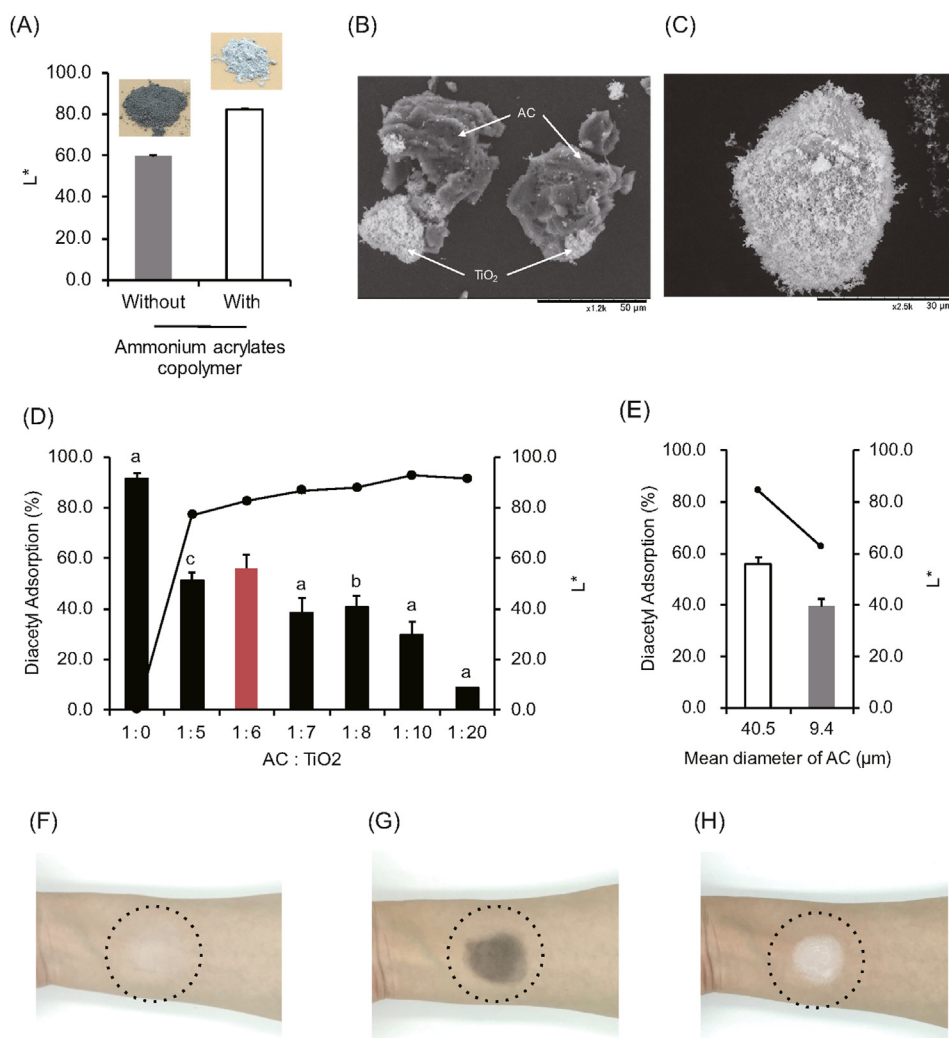
### 3.3. Development of WAC

ACs are difficult to use as a cosmetic ingredient applied on human skin, because of its black color. To use the AC (carbon A) in deodorant products, it was necessary to change the AC color to white. TiO<sub>2</sub> is an effective light-scattering material, due to its high refractive index [22]. Thus, to obtain the white color, AC was mixed with TiO<sub>2</sub>. However, the L\* value was found to be 62.5, which corresponded to a gray AC (Figure 4A). In addition, the scanning electron microscope (SEM) images revealed that TiO<sub>2</sub> did not completely adhere to the AC (Figure 4B).

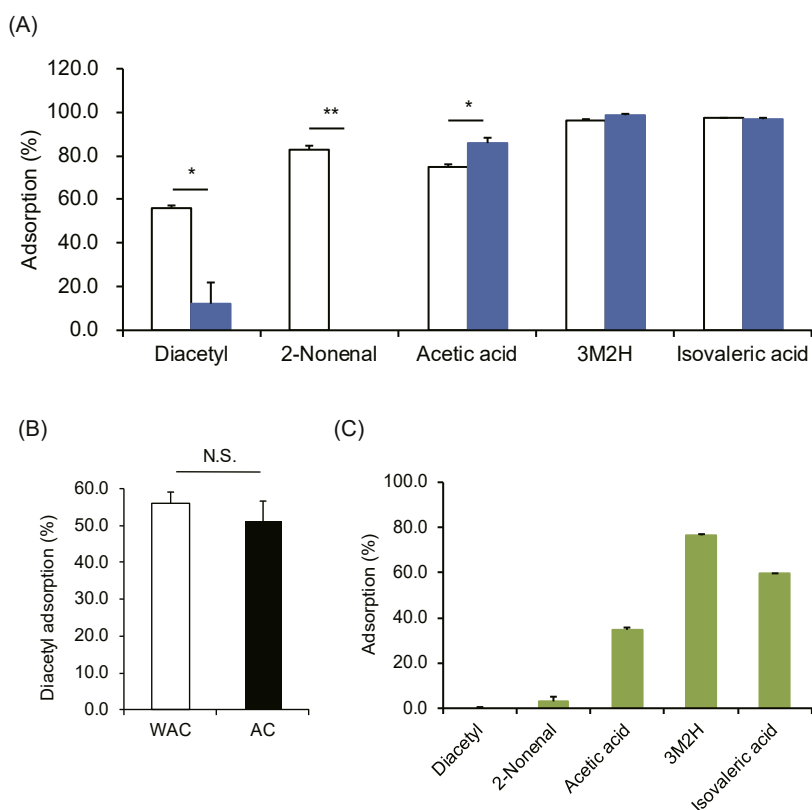
To enhance the adherence of TiO<sub>2</sub> to the AC, it was coated with a blend of TiO<sub>2</sub> and an ammonium acrylates copolymer solution. This resulted in an almost white WAC with a higher L\* value compared to that of the blend without the ammonium acrylates copolymer (Figure 4A). Additionally, the SEM images showed that the AC was fully coated with TiO<sub>2</sub> as a result of the incorporation of ammonium acrylates copolymer

(Figure 4C). The ammonium acrylates copolymer has been used as an adhesive for mascara, eyeliner, and eyelash extensions and as a film-forming agent for nail and skincare products [23, 24]. The ammonium acrylates copolymer was first coated on the AC surface before adding TiO<sub>2</sub>. The SEM result, which indicates the complete coating of AC with TiO<sub>2</sub>, can be attributed to the effect of the ammonium acrylates copolymer as a bonding agent. On the other hand, coating AC with ZnO and ammonium acrylates copolymer resulted in blue and black colored material, which was not suitable for use in leave-on products (data not shown).

Next, the appropriate quantities of AC (mean diameter = 40.5 μm) and TiO<sub>2</sub> were determined by analyzing the adsorption abilities and L\* values of samples with different AC:TiO<sub>2</sub> ratios. Increasing the TiO<sub>2</sub> content resulted in increased the L\* and decreased the adsorption levels of diacetyl (Figure 4D). This indicates that higher levels of TiO<sub>2</sub> blocked the AC pores. Considering both the tendency for adsorption and L\* value, the most appropriate ratio of AC to TiO<sub>2</sub> in the WAC was determined to be 1:6. Using this ratio, smaller AC (a mean diameter of 9.4 μm) was used to fabricate WAC. However, results indicated a lower adsorption level and L\* value for 9.4-μm mean diameter than that for 40.5-μm mean diameter (Figure 4E). WAC produced using this ratio had an acceptable appearance on the skin (Figure 4F), unlike the black color of AC (Figure 4G) and unnatural tone of only TiO<sub>2</sub> (Figure 4H). These results demonstrate that appropriate WAC consists of AC (a mean diameter of 40.5 μm)/TiO<sub>2</sub> blend 1:6 and ammonium acrylate copolymer. Some studies of TiO<sub>2</sub>/AC composites have been reported [25, 26, 27, 28].



**Figure 4.** Efficacy evaluation of the WAC prepared with various ratios of AC and TiO<sub>2</sub>. (A) The difference in the L\* values of the WACs with and without ammonium acrylates copolymer. (B and C) Scanning electron microscope images of WAC prepared (B) without and (C) with ammonium acrylates copolymer. (D) Adsorption (bar) and L\* value (line) of each WAC. Diacetyl was used as a representative odor compound. (E) Difference of diacetyl adsorption level (bar) and L\* value (line) in WAC induced by AC with a mean diameter of 9.4 and 40.5 μm. The ratio of AC to TiO<sub>2</sub> in each WAC is 1:6. (F–H) human skin images after the application of (F) WAC, (G) AC, and (H) TiO<sub>2</sub>. All results are from three independent experiments and expressed as means ± standard deviation, where n = 3. a: p < 0.05; b: p < 0.01; and c: not significant at a ratio of 1:6 of AC:TiO<sub>2</sub> (by one-way ANOVA followed by Dunnett's test).



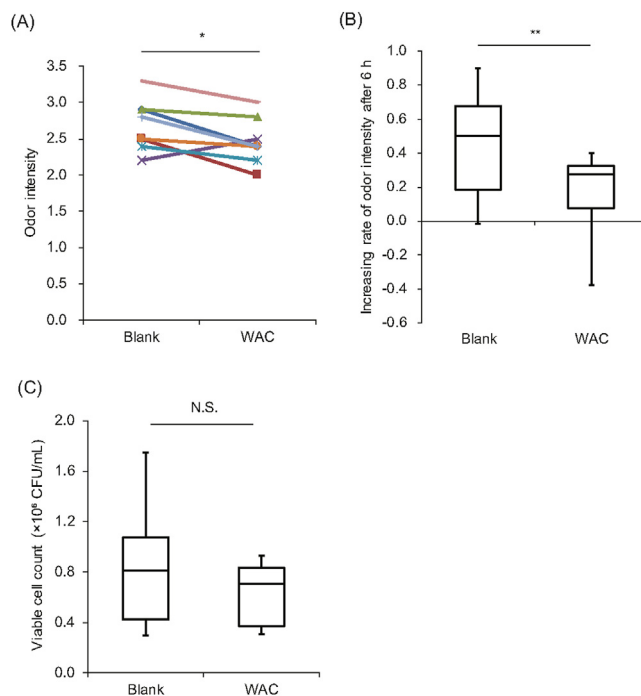
**Figure 5.** Adsorption abilities of WAC toward body malodor compounds. (A) Adsorption levels by WAC (white bar) and ZnO (blue bar). (B) Comparison between the diacetyl adsorption levels of WAC (white bar) and AC (black bar) using the same absolute content of AC and WAC. (C) Adsorption levels of various VOCs by TiO<sub>2</sub>. Data are shown as three independent experiments and expressed as means  $\pm$  standard deviation, where  $n = 3$ . N.S.: Not significant; \*:  $p < 0.05$ ; and \*\*:  $p < 0.01$  (by Welch's t-test).

These studies have focused on the application of TiO<sub>2</sub> as a photocatalyst owing to its high activity, which decomposes various organic compounds in air and water [25, 29, 30]. TiO<sub>2</sub> forms three polymorphs: anatase, rutile, and brookite. Generally, anatase polymorph exhibits higher photocatalytic activity than other forms [31]. However, TiO<sub>2</sub> for WAC development is selected based on the rutile form and primary grain size of the pigment particles (mean diameter of 0.25  $\mu\text{m}$ ) that possess high hiding power. This is because TiO<sub>2</sub> for WAC does not cause any risk to human skin and health and WAC facilitates a natural color tone to the human skin.

### 3.4. *In vitro* adsorption effect of WAC on the body malodor compounds

The adsorption ability of WAC was evaluated toward five body malodor compounds. The adsorption level of WAC was found to be significantly higher compared to that of ZnO for both diacetyl and 2-nonenal. However, WAC showed slightly lower adsorption effect compared to that of ZnO for acetic acid (Figure 5A). In contrast, no significant difference was found between the adsorption ability of WAC and ZnO for 3M2H and isovaleric acid. To compare the adsorption abilities of AC and AC contained in WAC, the adsorption level in the same amount of AC was tested using diacetyl, which could not be adsorbed on TiO<sub>2</sub>. It was found that the adsorption ability of WAC did not significantly change compared to that of AC (Figure 5B). This result demonstrated that the pores of WAC were not completely blocked by TiO<sub>2</sub>. Thus, WAC exhibited better deodorant characteristics compared to the control (ZnO).

Previous studies have reported that TiO<sub>2</sub>/AC composite photocatalysts enhance adsorption level toward VOCs than AC alone [27, 32]. Our rutile TiO<sub>2</sub> exhibited adsorption levels of more than 60% toward 3M2H and isovaleric acid (Figure 5C). However, diacetyl and 2-nonenal were not adsorbed on TiO<sub>2</sub> (Figure 5C). Moreover, WAC maintained the adsorption ability toward diacetyl than AC alone (Figure 5B). These results suggest that similar to the case of ZnO, short-chain fatty acids can be adsorbed on TiO<sub>2</sub>, rather than decomposition by TiO<sub>2</sub> photocatalyst.



**Figure 6.** *In vivo* evaluation of the suppression of axillary odor by WAC. (A) Suppressive effect of WAC on axillary odor generation after 6 h of sample application. Each marker represents one subject ( $n = 8$ ). \*:  $p < 0.05$  (by paired t-test). (B) Differences between the increase rate of axillary odor intensity in the blank and WAC application cases. The increase rate was calculated as the odor intensity difference between the start time of the test and after 6 h of WAC application. Data are shown as means  $\pm$  standard deviation;  $n = 8$ . \*\*:  $p < 0.01$  (by paired t-test). (C) Changes in viable cell counts ( $\times 10^6$  CFU/mL) of the axillae after the WAC application. Bacteria were collected from each axilla after 6 h of WAC application. Data are shown as means  $\pm$  standard deviation;  $n = 8$ . N.S.: Not significant (by paired t-test).



### 3.5. Suppression of axillary odor generation *in vivo* using WAC

The suppression effects of WAC on the generation of axillary odor were confirmed *in vivo* using a spray sample of WAC. After 6 h of the sample application, the increasing in the axillary odor intensity was significantly suppressed in the subjects that used WAC compared to the control that did not apply the product (Figure 6A and B).

Bioluminescence assay is one of the various assays for viable cell counts, measuring the intracellular ATP concentration as an indicator of cell viability [33]. This is a widely accepted, easy, and rapid assay to determine the microbial counts in foods and the environment [34, 35, 36]. To confirm the changes in the viable microbe count as a result of the WAC application, the intracellular ATP value was converted to a viable cell count of *S. epidermidis*, which is one of the major resident skin bacteria. No significant difference was observed in the viable bacterial count between the body parts treated and untreated with WAC (Figure 6C). These results demonstrate that the suppression effect of WAC on the generation of axillary odor via deodorizing function and not bactericidal effects.

## 4. Conclusions

WAC was developed as a novel deodorant material that can be applied to the human skin. Various key malodor compounds emitted from different body parts were successfully adsorbed on the proposed material. Moreover, the material can effectively suppress axillary odor generation. Thus, the proposed deodorant material could be considered a potential alternative to the deodorants available commercially. Moreover, our WAC can be applied to an interior wall material in the room, which is a requirement when painting using a white color, to prevent the generation of VOCs. However, the process will need to be scaled up and transformed into a continuous process for the production of deodorants. WAC can also provide psychological relief to individuals struggling with unpleasant body odor. We believe that WAC will be widely applied in the field of cosmetics, pharmaceuticals, and environmental industry.

## Declarations

### Author contribution statement

Takeshi Hara: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Haruna Nabei: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

Ayumi Kyuka: Conceived and designed the experiments; Performed the experiments.

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### Competing interest statement

The authors declare the following conflict of interests: [All authors are employees of Mandom Corp, which funded this research. Mandom Corp played no role in this study design, data acquisition, or the decision to publish.]

### Additional information

No additional information is available for this paper.

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