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# Physicochemical properties, antimicrobial activity, and contaminants in personal care products from Bangladesh

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## **ABSTRACT**

Personal care products help maintain hygiene and appearance by cleansing and protecting the skin and hair, supporting overall health and barrier function. The research was aimed at evaluating the quality of different locally available skin care products. This study assessed the physicochemical properties, antimicrobial activity, and the presence of Na+ and K+ ions in various personal care products (PCPs), including face washes, shower gels, and shampoos available in Dhaka, Bangladesh. It also evaluated the total active ingredient content, saponification, acid, and iodine values, the presence of plastic microbeads, and heavy metals such as lead (Pb) and cadmium (Cd). Antibacterial susceptibility tests against Escherichia coli (E. coli) and Staphylococcus aureus (S. aureus) were conducted using the Agar Diffusion Method. Na+ and K+ ions were quantified using a flame photometer. Active ingredient levels, saponification, acid, and iodine values were determined through titrimetric methods. The shapes of plastic microbeads were examined with a stereo-microscope, and their composition was analyzed spectrophotometrically. Heavy metals were measured using atomic absorption spectrometry (AAS). The pH of the samples ranged from 4.62 to 10.23. Shampoos exhibited the highest antimicrobial activity, while face washes were the least effective. Na+ concentrations varied from 49.45 to 218.74 mgL<sup>-1</sup>, and K<sup>+</sup> levels ranged from 1.3 to 125.60 mgL<sup>-1</sup>. Saponification values were in between 160.0 and 700.0 mgg<sup>-1</sup>, and acid values ranged from 0.9 to 11.0 mgg<sup>-1</sup>. Microbeads were either spherical or irregular, composed mainly of polyamide, polyethylene, and polyester. Lead concentrations varied between 0.75 and 7.54 mgL<sup>-1</sup>, with face washes containing high levels. Cadmium was below detection limits. The study highlights the need for careful selection of PCPs, considering their potential adverse effects on skin health and hygiene.

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## 1. Introduction

Personal care products (PCPs) help maintain hygiene and physical appearance by protecting against diseases (Kumar et al., 2021). They shield the skin from microbial attacks and repair damage (face wash or shower gel) or improve hair strength and prevent breakage (shampoo) (Karnwal et al., 2023). Cleansing the skin is essential for keeping it clean, healthy, smooth, and glossy, which is crucial for maintaining skin integrity and barrier function (Solanki et al., 2020). Hair care products clean the hair of sebum and scalp debris, improve hair fiber structure, and stimulate hair follicles and scalp (Fernandes et al., 2023). Face washes and shower gels have similar ingredients like surfactants and therapeutic agents, such as antimicrobials (triclocarban and triclosan), antiinflammatory agents (Vitamin B<sub>3</sub>), antioxidants (Vitamins A, C, and E), gelling agents (Carbopol 940), preservatives (methyl paraben, propyl paraben), and humectants (hyaluronic acid, propylene glycol) (Hayati et al., 2020). Shampoos mainly contain surfactants (cationic or anionic), co-surfactants (cocamidopropyl betaine), pH adjusters (citric acid), viscosity boosters (sodium chloride), and preservatives (El-Khordagui et al., 2021). The cleansing action is linked to micelle formation, which helps oily materials form stable emulsions (lather) that are easily washed away with water (Pathak et al., 2021).

Healthy skin maintains an optimal pH range between 5.4 and 5.9, which is crucial for sustaining the skin's natural bacterial flora, essential for protecting against harmful pathogens and maintaining overall skin health. However, a significant number of facial cleansers available in the Bangladeshi market fail to disclose their pH values, which could potentially affect their safety and effectiveness (Tarun et al., 2014). The electrical conductance of skin care products is a critical indicator that provides insights into the presence of alkalis, acids, or free ions within the product, helping to assess its compatibility with skin's natural properties (Shcherbakov et al., 2021). Products endowed with antibacterial properties not only offer a shield against microbial infections but also play a pivotal role in retaining the skin's moisture. They revitalize the skin by facilitating the removal of dead cells from the surface, thereby promoting a healthier and more youthful appearance. This process is akin to how hair care products function for the scalp, ensuring cleanliness and nourishment (Kang, et al.,

2022). The presence of sodium ions (Na<sup>+</sup>) in personal care products (PCPs) can be attributed to various sources such as electrolytes, pH regulators, and ionizable chelating agents. Products incorporating potassium hydroxide (KOH) as a pH stabilizer are notably rich in potassium ions (K<sup>+</sup>), which contribute significantly to the product's overall ionic content. The inclusion of minerals in PCPs is imperative as they impart a healthy, emollient glow to the skin, enhancing its texture and appearance (Michalak et al., 2021). The efficacy of shampoos in cleansing is largely dependent on the type and concentration of surfactants employed in their formulation. These surfactants vary according to the specific requirements of different hair types, thereby ensuring targeted care and treatment (Bezerra et al., 2023). The active ingredient content in shampoos, typically quantified as the percentage of surfactant like sodium lauryl ether sulfate (SLES) or its equivalents, is a vital measure that determines the cleansing power of the product (Ziółkowska et al., 2021). Parameters such as saponification value, acid value, and iodine value are indispensable in evaluating the quality of PCPs that contain fatty substances like oils. These metrics provide a comprehensive understanding of the chemical properties and stability of the product (MacArthur et al., 2021).

Microbeads, composed of synthetic polymers such as polyethylene, are minuscule plastic particles, usually less than 5 mm in diameter. They are predominantly used in PCPs to enhance the product's cleansing or exfoliating functions, serving as effective application enhancers (Bikiaris et al., 2024; Ghosh et al., 2021). The physical forms of microplastics, whether round, spherical, or irregular, contribute to the textural properties of the products in which they are incorporated (Rosal et al., 2021). However, numerous studies have highlighted the alarming environmental and health risks posed by the release of plastic microbeads into ecosystems (Tohura et al., 2025). These microplastics not only disrupt marine life but also have the potential to cause severe health issues when ingested by humans (Ziani et al., 2023). The long-term use of PCPs can lead to the accumulation of trace heavy metals in the human body, a concern that has garnered considerable attention due to its implications for human health (Balali-Mood et al., 2021). Metals such as Pb, Cd, Ni, and Zn are commonly found in PCPs. Regulatory bodies like the FDA's Cosmetic Ingredient Review Expert Panel have established limits for the permissible concentration of these metals in cosmetic products, with the maximum allowable concentration for lead being 20 mg/L. Meanwhile, the World Health Organization (WHO) recommends a stricter limit of 10 mg/L for lead in consumer products (Raza-Naqvi et al., 2022; Abed et al., 2024). The presence of these metals in PCPs is linked to various health problems, including cancer, kidney damage, and adverse effects on the reproductive and renal systems, underscoring the need for stringent quality control and regulatory oversight in the formulation of personal care products (Charkiewicz et al., 2023). The objectives of the present work can be outlined as follows: (i) Assessment of different physicochemical properties such as pH, EC, and solubility of three types of PCPs (ii) Antimicrobial activity of samples against Escherichia coli and Staphylococcus aureus. (iii) Quantification of Na+ and K+. (iv) Total active ingredient content in shampoo. (v) Determination of some important parameters in pursuit of examining quality of skin care products including saponification number, acid value, and iodine value. (vi) Determination of plastic microbeads and their characterization by both visually with microscope spectrophotometrically by the FT- IR spectrum and (vii) Analysis of samples for Pb and Cd.

#### 2. Materials and methods

#### 2.1. Sample collection

Three different PCPs (face wash, shower gel, and shampoo) of imported and indigenous brands were collected in October 2021 from different supermarkets of Dhaka city, Bangladesh. The samples were coded as  $P_1$ - $P_7$  (face wash, n=7),  $P_8$ - $P_{13}$  (shower gel, n=6) and  $P_{14}$ - $P_{20}$  (shampoo, n=7) along with their necessary information (Table 1). All of the samples were stored at room temperature (25-30 °C) until the analysis was carried out.

#### 2.2. Instruments, apparatus and reagents

Instruments used in this analysis were pH meter (Model no: HI 2211, HANNA instruments, USA), a conductivity meter (Model no: HI 2211, HANNA instruments, USA), stereo or dissecting microscope (EUROMEX Holland, NZ.1902P), Laminar air flow systems equipped with HEPA (High-Efficiency Particulate Air), Flame photometer (JENWAY PFP 7), and Atomic Absorption Spectrophotometer (Varian AA240, Australia). The reagents were analytical grade *n*-Hexane (RCI Lab scan Limited, USA), Methanol (Merck KGaA, Darmstadt, Germany), DCM (Merck KGaA, Darmstadt, Germany), Alcoholic KOH, Aqueous KOH, and Deionized water.

#### 2.3. Physicochemical parameters

The samples were weighed accurately  $5.0 \pm 0.01$  g by analytical balance and placed in a 100 mL beaker. Distilled water (45 mL) was added and samples were made dispersed in it and allowed to settle at room temperature. pH probe of previously calibrated pH meter was inserted in the sample and hold for a few minutes to get a stable reading. For the determination of EC, the electrode of the EC meter was calibrated with 0.1 M standard KCl and conductivity of each sample was measured. For solubility test, one or two drops of samples was added to 0.5 mL of distilled water in a test tube. The tube was tapped with finger to mix or stir gently with a glass rod. The sample was recorded as soluble or insoluble in distilled water. The process was repeated in the same manner with n-hexane, dichloromethane (DCM) and methanol.

## 2.4. Quantification of Na<sup>+</sup> and K<sup>+</sup>

Each sample (5 mL) was transferred into 50 mL volumetric flask and was made up to the mark with distilled water. Standard solution (1000 mgL $^{-1}$ ) was diluted to 100 mgL $^{-1}$ . Standard solutions of 10, 20, 30, 40, and 50 mgL $^{-1}$  were prepared from stock solutions of both Na $^{+}$  and K $^{+}$ . At first, flame photometer was allowed to give a sufficient warm-up period to ensure aspirate demineralized water between samples to clean out the sample tube and aspiration. The peak reading was set according to the instrument instructions using the standard solution. The emission intensity of each standard solution and each sample solution was measured. The accuracy and reproducibility were checked by measuring the standards several times.

## 2.5. Determination of active ingredient

Cationic solution of Cetyltrimethylammonium bromide was prepared firstly which was standardized against primary standard anionic solution later on. Each sample of shampoo was weighted accurately to give approximately 0.320 g of combined  $SO_3$  into a 250 mL beaker. 700 to 800 mL of warm distilled water was transferred to a 1L volumetric flask. It was warmed on steam bath and shaken gently until the sample was dissolved. The solution was diluted and mixed thoroughly. 10 mL of the sample solution was pipetted out into a 100 mL glass stoppered cylinder. 25 mL of methylene blue solution and 10 mL of chloroform was added into it. It was titrated with cationic solution to the correct end point.

Table 1. Information about different brands of collected PCPs samples

ID	Types of PCPs	Batch No.	Manufacturing date	Expiry Date	Net Wet
$P_1$	Oil-free face wash with menthol	61009	1-02-21	1-01-24	100 mL
$P_2$	Blackhead clearing facial scrub	66	25-09-21	24-09-24	100 g
$P_3$	Anti-pimple face wash	79U080	20-03-21	19-03-24	50 g
$P_4$	Deep cleaning men's face wash	81387	1-03-21	1-03-24	100 mL
$P_5$	Brightening men's facial scrub	314	5-01-21	5-01-24	50 g
$P_6$	Facial cleanser	1507	1-06-21	1-06-24	100 mL
$P_7$	Oil control herbal face wash	0212100022	1-04-21	1-03-24	100 mL
$P_8$	Gentle scrub shower gel	400/39	10-12-20	11-10-23	250 mL
$P_9$	Men's body cleanser	10235690	20-01-21	20-06-23	250 mL
$P_{10}$	Aroma therapy sensual shower gel	1122	20-03-21	19-03-23	250 mL
$P_{11}$	Antibacterial body wash	68339594	27-03-21	27-09-23	300 mL
$P_{12}$	Men's shower gel with aroma	04/02	5-05-21	5-04-24	250 mL
$P_{13}$	Men's body foam	G0212599	16-09-20	16-09-22	250 mL
$P_{14}$	Commercial shampoo	31	30-01-21	29-01-23	375 mL
$P_{15}$	Anti-dandruff shampoo	11	20-04-21	19-04-23	180 mL
$P_{16}$	Commercial shampoo	0040930263	1-02-20	1-01-23	180 mL
$P_{17}$	Commercial shampoo	11	23-05-21	22-05-23	170 mL
$P_{18}$	Anti-dandruff shampoo	0317930266	1-11-20	1-10-23	180 mL
$P_{19}$	Pediatric shampoo	400/39	24-03-21	1-03-24	200 mL
$P_{20}$	Professional shampoo	32	6-07-21	5-07-23	185 mL

## 2.6. Determination of saponification, acid and iodine values

1.0-2.0 g of sample was weighed into a 250 mL conical flask and 25 mL of KOH (alc.) was added to it. The mixture was refluxed for at least half an hour over a water bath to ensure the complete saponification. The contents of the flask were cooled and the unreacted alkali was titrated with standard 0.5 M HCl solution using phenolphthalein as indicator. A blank experiment was done under identical conditions with omission of sample. The difference between the volumes of HCl required in the two titrations corresponded to the amount of alkali used in the saponification. For acid value, about 0.5 g of sample was weighed in a beaker, transferred into a conical flask, dissolved in approximately 30 mL ethanol and titrated with a standard 0.01 M KOH solution using phenolphthalein indicator. The volume of KOH required for the titration was recorded. For iodine value, about 0.5 g of the sample was weighed in a 250 mL conical flask and 10 mL of carbon tetrachloride was added to dissolve the sample followed by 25.0 mL of Hanus solution. 100 mL of water was poured followed by 10 mL of 12% KI solution. The iodine left unreacted was titrated with previously standardized 0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution using starch indicator. A blank experiment was performed with the omission of sample. The difference between these two volumes of thiosulphate solution gives the volume of thiosulphate required to titrate iodine absorbed by the sample.

## 2.7. Antimicrobial assay

Susceptibility of the *E. coli* and *S. aureus* isolates to different antimicrobial agents was measured in *vitro* by employing Agar Diffusion Method by Kirby-Bauer, 1966. In this method, a suspension of test organism (*E. coli* and *S. aureus*) was prepared in normal saline (0.85% NaCl) to match the equivalent turbidity standard to that of MacFarland 0.5 standard. A sterile cotton swab was dipped into the suspension. The medium of choice was Mueller-Hinton agar which was poured into plates to a uniform depth of 5 mm. The swab was then heavily inoculated over the entire surface of the plate to obtain a confluent growth of the organism. The samples were applied aseptically to the surface of the inoculated plates. The plates were then inverted and incubated at 37°C for 24 h. After incubation, diameters of the zone of inhibition were measured in mm.

#### 2.8. Plastic microbeads in samples

For analysis of plastic microbeads in samples, pre-treatment of the samples was done by dissolving 0.5 to 3.0 g sample in 200 mL warm deionized water in a beaker. The solution was passed through a 5 mm screen, and the filtrate was collected in a conical flask. The filtrate was poured into a 0.05 mm stainless steel filter. The filter containing microbeads was removed horizontally, and the filter was placed on an aluminum foil disc. The aluminum foil disc was placed in an oven at 50-60 °C for about 30 minutes to remove residual moisture. The dry solid microbeads were analyzed by FT-IR spectrometer. Before the analysis, the instrument was calibrated with standard test pieces using the procedures provided by instrument manufacturer. The finely ground sample was mixed with powdered potassium bromide and pressed under high pressure. Under pressure, the potassium bromide melted and the sample was sealed in a matrix. The resulting KBr pellet is inserted in the instrument. The instrument was scanned from 400 to 4000 cm<sup>-1</sup>. The sample was later stored in a dry cabinet for later analysis and observed visually under stereo-microscope.

## 2.9. Heavy metal (Pb and Cd) analysis

Heavy metals determination in PCPs was done using AAS. 10-15 mL of each sample was taken in a moisture dish and placed in oven at 105° C for 3 hours. 2 g of dried sample was weighed accurately in a 100 mL beaker and 10 mL of conc. HNO3 was added to it. The beaker was placed at 95°C for 15 min. The digested sample was cooled and 5 mL of conc. HNO3 was added followed by heating for additional 0.5 hour at the same temperature. The process was kept repeating until the solution being reduced to about 5 mL. The sample was cooled again and 2 mL of deionized water was added into which 3 mL of 30% H<sub>2</sub>O<sub>2</sub> was poured. The sample was heated gently to start the peroxide reaction until the effervescence was subsiding. Then, 5 mL of conc. HCl and 10 mL of deionized water were added. Later on, the sample was heated at 95 °C for 15 min without boiling. The sample was cooled and diluted to 50 mL. The samples were analyzed using a specific hollow lamp of Lead ( $\lambda$ =217.0 nm), and Cadmium ( $\lambda$ =228.8 nm). Samples were inhaled using a nebulizer, and absorbance was measured using a blank as a reference.

#### 3. Results and discussion

## 3.1. Physicochemical parameters

All face wash and shampoo samples were soluble in distilled water and insoluble in n-hexane, DCM, and methanol. The same trend was found in shower gel, except P9 and P12 which were soluble in n-hexane. Out of 20 analyzed PCPs, the pH in face wash (P1-P7), shower gel (P<sub>8</sub>- P<sub>13</sub>) and shampoo (P<sub>14</sub>-P<sub>20</sub>) were 5.80-10.23, 4.62-9.46, and 5.75-7.19, respectively (Fig. 1a). It demonstrated that PCPs could be acidic or basic depending on manufacturer, brand, and uses. Two face washes ( $P_2$  and  $P_7$ ) were acidic (pH<7.0) and five (P1, P3, P4, P5 and P6) were alkaline (pH>7.0). P2, P4 (normal facial cleanser), and P7 (herbal face wash) were pH balanced which helps skin's acid mantle secret naturally. P3 (anti-pimple men's face wash) and P<sub>5</sub> (oil control men's face wash) had pH>10.0, much higher than that of skin which could scrap acid mantle away making skin vulnerable to bacterial development (Tarun et al., 2014). Both P<sub>10</sub> and P<sub>12</sub> had their pH 4.62-6.0, which is balanced enough to keep acid mantle intact by maintaining skin's natural pH (Hawkins et al., 2021). P<sub>8</sub> and P<sub>13</sub> were found to have pH near neutral zone while P<sub>9</sub> and P<sub>11</sub> were alkaline (7.90-9.46). As alkaline pH is accompanied by a slightly enhanced trans epidermal water loss, it could damage the acid mantle functioning as an antibacterial barrier, allowing the entry of potential irritants and allergens (Lukić et al., 2021). All the 7 samples of shampoo, had pH 5.75-7.19 which is much higher than hair shaft pH 3.6 and even higher than the scalp pH 5.5 indicating all of the shampoos could cause friction, frizz, hair breakage and enhance hair tangling (Rahma and Lane, 2022). P<sub>15</sub> and P<sub>18</sub> were anti dandruff shampoo. P<sub>19</sub>, claimed to be a pediatric shampoo, contained citric acid (as labeled) which acts as pH regulator (D'Souza and Rathi, 2015). P20 was claimed to be a lower sulphatebased shampoo. But the observed pH 6.95 did not really agree with this (Sastrawidana et al., 2019).

Out of 20 analyzed PCPs samples, the EC was 5010-1324, 9370-3560 and 6710-2546 μScm<sup>-1</sup> for face wash, shower gel and shampoo, respectively (Fig. 1b). Among the 7 face washes, P1 showed highest value of conductance. P6 was found to have the lowest conductance due to the absence of free ions. Among 6 shower gel samples, P11 had the highest value of conductance and P9 had the lowest. The conductance of rest of the products varied in between them depending upon the availability of free ions. The conductivity in shampoos is mainly due to the presence of Na+ ion as chelating agent in the form of salt or surfactant. P18, an antidandruff shampoo containing NaCl as one of its main ingredients exhibited the highest electrical conductance. Second large value was seen for P<sub>15</sub> which also is an anti-dandruff shampoo. Other shampoos showed conductivity in the range between 3100-5200 µS with an exception of P19, a pediatric shampoo with conductivity of 2546 μScm<sup>-1</sup> ,contains disodium EDTA as a minor ingredient.

## 3.2. Na<sup>+</sup> and K<sup>+</sup> content

 $P_5$  was found to contain highest amount of  $Na^+$  ions (Fig. 1c) among the samples of face wash probably due to the presence of NaCl, sodium benzoate,  $Na_2SO_4$  together with disodium EDTA as major ingredients. Sample  $P_6$  contained sodium laureth sulfate and sodium lauryl sarcosinate as leading sources of  $Na^+$  ions. Samples ( $P_1$  and  $P_7$ ) containing chelating agent like disodium EDTA in slightly lower concentration had moderate amount of  $Na^+$  ions.  $P_4$  contained NaOH and NaCl as principal sodium containing compounds. Average content of Na in the face wash samples was observed to be 117.17 mgL $^-$ 1. Among all the samples of shower gel,  $P_{10}$  and  $P_{11}$  were observed to contain the highest amount of  $Na^+$ . Samples  $P_8$ ,  $P_9$ , and  $P_{12}$  showed moderate value for  $Na^+$  content.  $P_{13}$  contained the lowest amount of  $Na^+$  ions. Average content of Na in the shower gel samples was 158.55 mgL $^-$ 1.  $P_{17}$  and  $P_{19}$  had the highest quantity of  $Na^+$  ions (among shampoo samples) as they

contain higher percentage of surfactants as well as chelating agents in their formulation. In  $P_{15}$ , sodium laureth sulphate, sodium salicylate, and sodium chloride along with sodium hydroxide could be the main sources of Na $^+$  (according to the ingredients mentioned in the packaging). All of these compounds were included in their formulation but in minute amount thus making the content of Na $^+$  ion minimum among 7 test samples of shampoo. Average Na $^+$  ion content in these samples was found to be 83.469 mgL $^-$ 1.

Out of 20 analyzed PCPs samples, the content of K+ was found in the range (1.33-92.63 mgL<sup>-1</sup>), (3.86-125.60 mgL<sup>-1</sup>), (1.33-6.40 mgL-1) for face wash, shower gel, and shampoo, respectively (Fig. 1d). P<sub>3</sub> contained K<sup>+</sup> in the highest quantity among samples of face wash as it was formulated with KOH. Sample P<sub>5</sub> also contained KOH but its potassium content was much lower than the former. Average amount of K content was found to be 29.23 mgL<sup>-1</sup>. Among samples of shower gel, P11 contained higher amount of K+ than other samples as it contained KOH along with KCl as buffering agent (Martin et al., 2024). P<sub>13</sub> had moderate amount of K<sup>+</sup> due to the presence of KOH as sole contributor of K<sup>+</sup> ions (according to the ingredients mentioned in the packaging). Average amount of K<sup>+</sup> in the samples of shower gel was 33.03 mgL<sup>-1</sup>. There was significant amount of KOH as pH balancer in the formulation of P19 as a pediatric shampoo (Ramer and Hinz, 2022). Rest of the shampoos were mainly surfactant-based shampoo, and not really pH balanced which was observed to be reflected in the identical value of potassium content exhibited by them.

## 3.3. Active ingredients in shampoo

According to Bangladesh Standards & Testing institution, minimum active ingredient content (percent by mass) as SLES (Sodium laureth sulphate) in shampoos should be 3. All tested shampoo samples complied with this requirement. Maximum percentage of SLES was found for  $P_{14}$  (17.10%) and minimum for  $P_{18}$  (13.05%) (Fig. 1e). Other shampoo samples had active ingredient ranging between 13.83-15.30%.  $P_{14}$  had maximum amount of sulphate-based surfactant, it has sodium laureth sulphate as main surface-active ingredient and can work effectively by lifting soil from the scalp (Leoty-Okombi et al., 2021).

#### 3.4. Saponification, acid and iodine values

Saponification value is important because it indicates the quality of the fat/oil used to produce the personal care product (MacArthur et al., 2021). Higher saponification value indicates more shorter carbon chain lengths of the fatty acids (Ivanova et al., 2022). Out of 20 analyzed PCPs samples, saponification value was found in the range (160.92-193.75 mgg<sup>-1</sup>), (277.44-395.35 mgg<sup>-1</sup>), (343.89-680.35 mgg<sup>-1</sup>) for face wash, shower gel, and shampoo respectively (Fig. 1f). Among the face washes, the highest saponification value was obtained for P2 and P5. The reason might be the presence of substantial content of saponifiable fatty materials like ascorbyl palmitate and acetate (in P2) as well as myristic acid (14-C length), stearic acid (18-C length), lauric acid (12-C) etc in it (MacArthur et al., 2021). All of the shower gel samples had higher values of saponification (250-400 mgg<sup>-1</sup>). Sample P<sub>10</sub> had only citric acid, it was found to have lesser saponification value. P14 contained citric acid (6-C) but did not contain any natural oil and the saponification value was moderate among shampoo samples. P<sub>16</sub> had the highest saponification value among shampoos due to the presence of 'Trihydroxystearin' besides citric acid. P<sub>19</sub> only contains citric acid as minor ingredient. Thus, it gave the lowest saponification number. Analysis of the acid value of the PCP samples showed that out of these 20 samples, the content of free acids was found in the range (0.91-10.59 mgg<sup>-1</sup>), (0.98-8.74 mgg<sup>-1</sup> 1), (0.43-2.29 mgg<sup>-1</sup>) for face wash, shower gel, and shampoo respectively (Fig. 1g). Sample P5 had the highest acid value due the presence of saturated fatty acids (myristic acid, stearic acid, and lauric acid) in their free forms. In case of shower gel, sample  $P_8$  had the highest acid value due the presence of same saturated free fatty acids mentioned above. Sample  $P_{10}$  exhibited the lowest acid value due to the presence of only one kind of fatty acid (citric acid). Analysis of the acid value of shampoo samples showed that,  $P_{19}$  had the highest acid value due to the presence of citric acid in its free form.  $P_{18}$  has the lowest acid value due to the lack of any kind of

unbound fatty acids. The rest of the samples had acid value which are almost similar. Hanus solution did not react with any of the samples under study and thus iodine value could not be obtained for the samples indicating an absence or very low level of unsaturated compounds under the tested conditions. Fatty materials present in all of the samples are in saturated form.

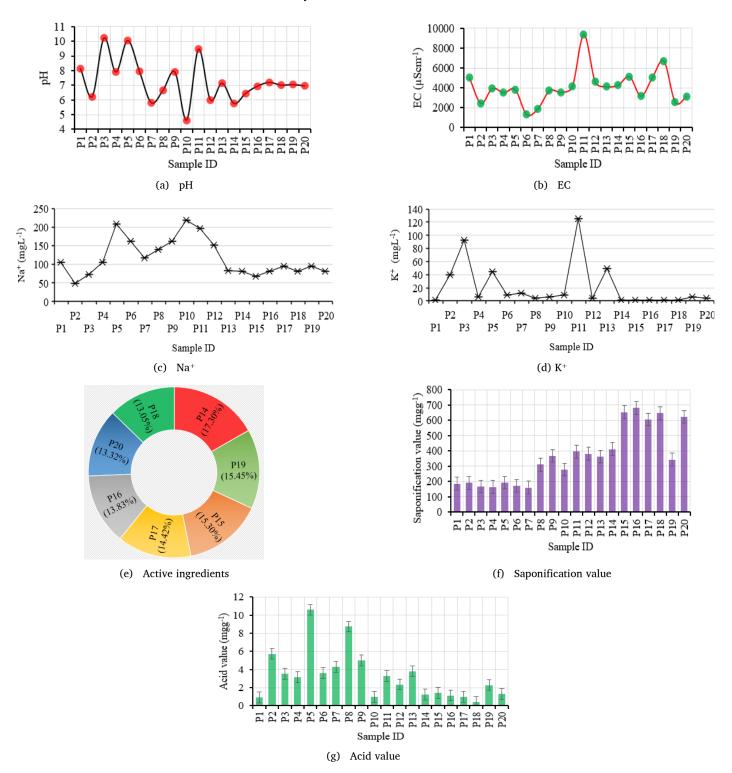


Fig. 1. Variations of different physicochemical parameters in the collected PCPs samples (a-g)

## 3.5. Antimicrobial assay

Inhibition zones greater than 10 mm were considered indicative of effective antibacterial activity, while zones of 6 mm were interpreted as no inhibition, as this corresponds to the

diameter of the control as well. All antimicrobial assays were performed in triplicate with a ±5% margin of error around the mean value. Analysis of the zone of inhibition showed that *E. coli* was completely resistant to P<sub>3</sub>, P<sub>4</sub>, P<sub>5</sub>, P<sub>6</sub>, and P<sub>7</sub> which was clearly revealed from inhibition diameter of 6 mm exhibited by them

(Table 2). P<sub>1</sub> and P<sub>2</sub> provided mild bacterial growth inhibition. P<sub>1</sub> had cocamidopropyl betaine as surfactant which shows great antibacterial activity, thus the sample created the largest area of inhibition against *E. coli* among all samples of face wash (Herrwerth et al., 2008; Ferreira et al., 2022). On the other hand, P<sub>2</sub> containing only SLS (sodium laureth sulphate) as antibacterial agent, provided a very slight inhibition. *S. aureus* was moderately susceptible to P<sub>4</sub> and P<sub>7</sub> as they were formulated herbally. Though sample P<sub>3</sub> claimed to be an anti-pimple face wash formulated with lemon fruit extract showed no detectable activity. The zone of inhibition depicted that no antibacterial activity had been detected in P<sub>8</sub>, P<sub>9</sub>, P<sub>10</sub>, P<sub>11</sub>, P<sub>13</sub> and very weak antibacterial activity had been observed in P<sub>12</sub> against *E. coli*. Sample P<sub>12</sub> had cocamidopropyl betaine, thus created the largest area of inhibition against *E. coli* among all shower gel samples (Fig. 2) (Herrwerth et al., 2008; Ferreira et al., 2022).

No antimicrobial activity had been detected in  $P_{11}$ ,  $P_{12}$  and  $P_{13}$  and minimal inhibition against bacterial growth was found to be provided by  $P_{10}$  while moderately strong resistance against bacterial growth was observed by  $P_8$  and  $P_9$  against S. aureus. Among samples of shampoo,  $P_{15}$  and  $P_{18}$  were shown to provide the most powerful guard against growth and development of both microbes under study. They both were anti-dandruff shampoos containing zinc pyrithione, SLS, and salicylic acids, as key ingredients which enabled them to show effective barrier against microbial activity (Umar et al., 2021). No antibacterial activity had been detected in the pediatric shampoo,  $P_{19}$ .  $P_{20}$ , a lower sulphate containing shampoo, exhibited lower anti-bacterial activity than other sulphate-based shampoos as the nitrogen-based bacteria is mainly killed by hydroxide ions present in alkaline formulations (Mawani et al., 2023).

#### 3.6. Analysis of plastic microbeads

Out of 20 samples of personal care products, 8 samples ( $P_1$ ,  $P_2$ ,  $P_3$ ,  $P_6$ ,  $P_7$ ,  $P_8$ ,  $P_9$ , and  $P_{13}$ ) were found to contain plastic microbeads (Table 3). The FT-IR band stretching and vibrational mode were identified by established literature (Hisam et al., 2024; Campanale et al., 2023; Kamnev et al., 2021). The detection range for the FT-IR was 400-4000 cm<sup>-1</sup>. The morphotypes of microbeads were detected to be round shaped, spherical, and rectangular but some were irregular shaped including filamentous, short stripes, and random fragments which were confirmed from their microscopic images (Fig. 3). Images were taken with a camera having a resolution of 3584 × 2746 pixels, with each image covering an area of 14.49 mm<sup>2</sup>.

The beads were mostly made of polyethylene which performs as abrasive whereas polyamide (nylon) acts as bulking agent. Overuse of microbeads in personal care products (PCPs) may pose a threat to the environment (Suemak, 2018). Due to their rough surface, beads have enhanced adsorption properties thus, toxic chemicals and heavy metals can be carried into human bodies causing acute health disorders (Wang et al., 2022; Ali et al., 2024). Several ASEAN nations are addressing microbeads in personal care products due to environmental risks. Thailand fully banned them in rinse-off cosmetics in 2020, while Indonesia is phasing them out. Malaysia and Singapore depend on voluntary industry action, and the Philippines has proposed but not passed any legislation. Vietnam, Brunei, Laos, Cambodia, and Myanmar are currently lack clear regulations (ASEAN Cosmetics Association, 2020).

Table 2. Zone of inhibition	on for PCP samples	against <i>E.coli</i> and <i>S.</i>	aureus
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ID	Zone of inhibition (mm)		ID	Zone of inhibition (mm)	
_	E. coli	S. aureus		E. coli	S. aureus
$P_1$	8	7	P <sub>11</sub>	6	6
$P_2$	7	6	$P_{12}$	7	6
$P_3$	6	6	$P_{13}$	6	6
$P_4$	6	9	$P_{14}$	13	20
$P_5$	6	6	$P_{15}$	24	25
$P_6$	6	7	$P_{16}$	6	20
$P_7$	6	9	$P_{17}$	12	20
$P_8$	6	18	$P_{18}$	24	24
$P_9$	6	10	$P_{19}$	6	6
$P_{10}$	6	7	$P_{20}$	10	11

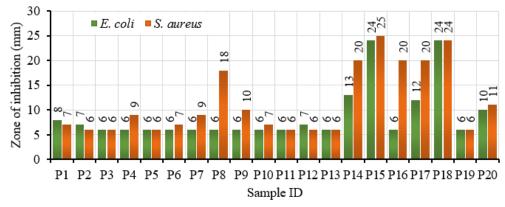
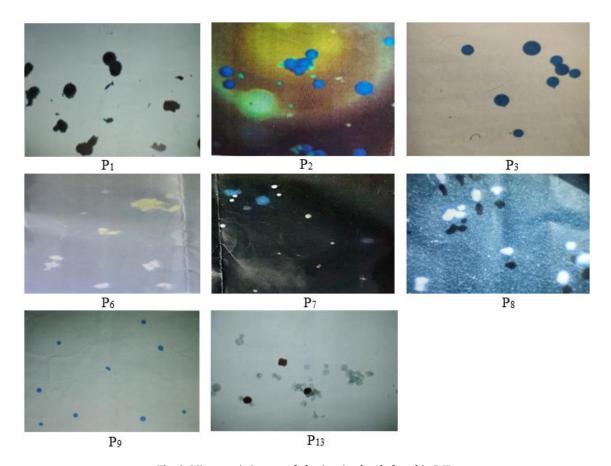


Fig. 2. Zone of inhibition for PCP samples against E. coli and S. aureus

Table 3. Absorption peaks in IR spectra for samples containing microbeads (Hisam et al., 2024; Campanale et al., 2023; Kamnev et al., 2021)

ID	Absorption (cm <sup>-1</sup> )	Composition of microbeads
$P_1$	3355 (broad N-H stretching), 2917 & 2855 (CH <sub>2</sub> asymmetric and symmetric st.), 2353 (CO <sub>2</sub> ), 1727 (C=O st. for N-C=O group), 1462 (CH <sub>2</sub> bending), 1164 & 1053 (C-O st)	Polyamide and Polyethylene
$P_2$	3395 (broad, N-H st.), 2916, 2855, 2352,728, 1635, 1545 &1465 (N-H bending and $CH_2$ bending), 723 (sp <sup>2</sup> C-H bending)	Polyamide and Polyethylene
$P_3$	3470 (broad, N-H st.), 2974, 2921, 2863, 2359, 1697 (C=O st), 1560 & 1373 (N-H bending and CH <sub>2</sub> bending)	Polyamide and Polyethylene
$P_6$	3435 (broad, N-H st.), 2919, 2850, 1468, 722	Polyamide and Polyethylene
$P_7$	3439 (broad), 2918, 2852, 2353, 1648, 1462 (CH <sub>2</sub> bending), 1063 (C-O st), 722 (sp <sup>2</sup> C-H bending)	Polyamide and Polyethylene
$P_8$	3401 (broad N-H st.), 2925, 2856 (CH <sub>2</sub> st.), 2368, 1713, 1637 (C=O st), 1403 (vibration of atomic ring)	Polyamide, Polyethylene and Polyester
$P_9$	3397 (broad, N-H st.), 2910, 2365 (CH <sub>2</sub> st.), 1637, 1431, 1371, 1241, 1162, 1062	Polyamide, Polyethylene and Polyester
$P_{13}$	3306 (broad, N-H st.), 2366, 2133, 1634	Polyamide and Polyethylene



 $\textbf{Fig. 3.} \ \ \textbf{Microscopic images of plastic microbeads found in PCPs}$ 

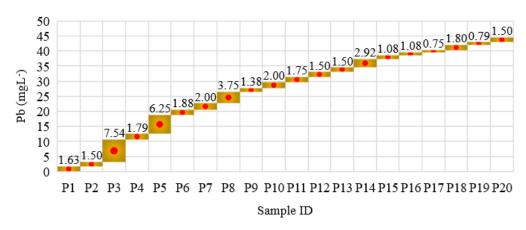


Fig. 4. Variation of Lead in different brands of PCPs

#### 3.7. Heavy metal analysis

The concentration of Pb varied significantly from one sample to another and also between different categories of PCPs. Notably all the 20 samples had Cd in below detection level. According to the Cosmetic Ingredient Review Expert Panel established by Food and Drug Administration (FDA) in the USA, the maximum allowable concentration of Pb in cosmetic products is 20 mgL<sup>-1</sup> (Abed et al., 2024). The World Health Organization (WHO) sets limit for Pb as 10 mgL-1 (Charkiewicz et al., 2023). All the samples complied with the WHO as well as FDA standards. Among face washes, lead content in P<sub>3</sub> and P<sub>8</sub> was relatively elevated compared to other PCPs. Among 6 shower gels, P8 had the highest Pb concentration which is still in the safe range. Other samples contained Pb in trivial amount which are below 2 mg/L. Among 7 shampoo samples, P14 had the highest Pb concentration and P<sub>17</sub> the lowest (Fig. 4). The detection of Pb in P19, a pediatric shampoo, at 0.79 mg/L is concerning due to children's heightened sensitivity to heavy metals. Even low levels of Pb exposure can impair neurological development. The Centers for Disease Control and Prevention (CDC) states that no blood lead level is considered safe for children. (CDC, 2025) Although the FDA recommends a 10 mgL-1 limit for lead in cosmetics (FDA, 2016), the presence of Pb in a child-focused product highlights the need for stricter regulation and monitoring.

Presence of Pb in shampoos can cause chronic headache and dizziness (Fiton et al., 2020). The detection limit for Pb and Cd was 0.35 and 0.85 mgL<sup>-1</sup>, respectively.

#### 4. Conclusion

This study evaluated various physicochemical and safety parameters of 20 personal care products (PCPs), including pH, antibacterial activity, Na+ and K+ ion content, active ingredients, saponification value, acid value, iodine value, presence of plastic microbeads, and heavy metals (Pb, Cd). Most samples (18 out of 20) complied with standard skin-care pH ranges, indicating suitability for topical use. Shower gels showed minimal antibacterial activity. Shampoos with lower sulfate content were generally less effective in terms of antimicrobial activity. Saponification values were moderately high across all PCPs. Samples with higher free saturated fatty acid content exhibited higher acid values. No iodine value was detected, suggesting an absence or minimal level of unsaturated compounds. No plastic microbeads were found in shampoo samples, which aligns with their non-exfoliating function. However, the potential risk of microbeads in other PCP categories remains a concern due to their ability to adsorb and transport toxic substances. Pb levels in all products were below permissible limits. Cd was not detected in any of the 20 samples. The study is limited by the number and regional availability of brands, and results may not reflect all products available in the national market. Regulatory bodies should enforce stricter quality control, particularly regarding labeling of active ingredients and heavy metal content.

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## **Conflict of interest**

The authors declare no conflict of interest in this research.

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