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Quality and Safety Aspects of Dried Scaly Stingray (*Himantura walga*) Fishery Product in Southern Regions of Bangladesh

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ABSTRACT

Fish drying is an old and well-established technique for generating nutritionally balanced human food. The present study examined the biochemical characteristics, quality and safety features of dried stingray products from major drying regions in coastal Bangladesh. Standard validated methods were applied to assess different compositions. Pesticide residues were analyzed using QuEChERS separation in conjunction with gas chromatography and mass spectrometry. The organoleptic properties revealed that dried fish samples were of good quality for human consumption and did not significantly (P>0.05) differ in all drying yards. The aerobic plate count (APC) for all products exceeded the regulatory limit, with the highest found ($1.2±0.01×10^6$ cfu/g) in Kuakata drying yard. Similarly, the highest total fungal load was found in the same yard ($1.5±0.02×10^3$ cfu/g). *Salmonella* spp., *Vibrio cholerae*, V*ibrio parahaemolyticus* and *Listeria monocytogens* were not detected in all dried products. Except for ash and pH content (p>0.05), the proximate components (protein, lipid, ash, moisture) and other spoilage metrics of dried stingrays recorded significant differences (P<0.05). The organochlorine residues (DDT and heptachlor) were not observed in all products, and no health risk was detected from the sampling stations associated with their pesticides. Based on biochemical compositions and the quality and safety properties, these dried fish products may be a realistic and safe food for human consumption.

INTRODUCTION

Bangladesh is blessed with an abundance of diverse fisheries resources **(Hanif *et al.*, 2015; Islam *et al.*, 2017)** and a wide variety of freshwater and marine fish species, including the shrimp and prawn, chinese pomfret, bombay duck, ribbon fish and silver jewfish used to produce dried fish and fishery products **(Paul *et al.*, 2018)**. In

Bangladesh, fisheries sector contributed to 3.57% and 25.30% of the countrys overall and agricultural gross domestic products (GDPs), respectively, and generated about $5 million in export revenue from fish and fishery products in 2017–2018, and only 14.74% of the nation's total fish production is derived from marine fisheries **(DoF, 2022)**. Almost 20% of Bangladesh's total marine catch is sun dried and supplied in the local markets **(Hasan *et al.,* 2016)**. Dried rays seafood products will play an important role in quality and safety for consumers both at home and abroad. Dried fish products have significant economic value and are the cheapest source of highly valued protein, lipids, vitamins, and minerals **(Siddique & Aktar, 2011)**.

The elasmobranch fisheries comprises of 24 species of rays **(Jit *et al.*, 2013)**, which are usually captured for their high-value fins, but the rays meat has some market value in the tribal area of Bangladesh **(Roy *et al.*, 2014)**. Stingrays meats are less expensive in local Bangladeshi markets than other marine fishes. Rays meats are generally dried in the sun and are used for human consumption, especially in the culinary soups and tannery industry in Bangladesh **(Hoq *et al.*, 2011)**. Traditionally, producing dried seafood products do not maintain the quality and safety aspects of the incredibly hygienic condition, low quality, improper procedure and elevated exposure to the sun during processing in the fish drying yards. Due to the prevention of contamination and other blow-fly infestation, different pesticides are commonly used at various stages of handling and processing to extend the freshness of stored dried products **(Chakma *et al.*, 2022)**. Keeping dried fish for an extended time may cause it to lose some of its nutritious values because of absorbing moisture from the surrounding environment **(Sultana *et al.*, 2010)**. Most traditional sun-dried products sold in local markets lack the adequate organoleptic properties for human consumption **(Reza, 2002; Hasan, 2006)** and also human activities is crucial in microbial contamination during different steps of processing **(Azam *et al.*, 2003a; Chakma *et al.*, 2020)**. Besides, the biochemical composition of dried fish is the crucial to the processors as it affects the physical and chemical qualities due to the preservation of the dried fish. Generally, dried fish product has a high nutritional value and contains essential components for a healthy and well-balanced body **(Koffi-Nevry *et al.*, 2011)**. Dried fishes are higher protein source which commonly used as a substitute for fresh fish when fresh fish is in scarcity **(Rahman *et al.*, 2017)**.

There have been a few studies on the quality of dried fish in Bangladesh, but they have all been limited to specific areas and types of fish. **Banna *et al.* (2022)** investigated on the nutritional composition, quality and safety of some dried fishes in Bangladesh. However, to our knowledge so far, no information has been reported on the nutritional quality and safety of dried scaly stingray (*H. walga*) collected from different commercial fish drying yards in Bangladesh. As the dried fishery products accessible in the local market is produced, there is an imperative need to frequently assess and review the reliability of dried fish produced in commercial drying yards in Bangladesh. The current findings may provide information on the proximate compositions, quality and safety aspects of dried stingrays, which may boost the suitability of dried products to consumers and also generating global markets. Therefore, the study intends to investigate the nutritional quality and safety aspects of dried stingrays from various commercial fish drying locations in Bangladesh, as well as vendors expectations of the products quality and perceived potential risks.

MATERIALS AND METHODS

Ethical statement

The experimental approach and regulations were used for research purposes and were approved (*PSTU/IEC/2022/44*) by the Institutional Ethical Committee (IES) of Patuakhali Science and Technology University (PSTU), Patuakhali, Bangladesh.

Study area

From three major fish drying yards, dried stingray (*Himantura walga*) were collected, viz. Kutubdiapara drying yard (Cox'sbazar), Banshkhali drying yard (Chattogram) and Kuakata drying yard (Patuakhali). The sampling locations are shown in Fig. (1).

Sample collection and preparation

About 3 kg of composites stingray (*H. walga*) were purchased triplicate from each of the three major fish drying yards from southern parts of Bangladesh. Insulated ice box was used to keep the sample, then labeling was done, and finally the samples were brought to the Seafood Quality, and Safety Laboratory at the Patuakhali Science and Technology University, Bangladesh. All the samples were kept at 4°C in refrigerator (SJ-VX79E-SL, Sharp, Japan) for further analysis.

Organoleptic characteristics

The organoleptic attributes such as color, odor, texture and insect infestation of dried stingray products were assessed by **Rasul *et al.* (2018)**, with slight modifications Table (1).

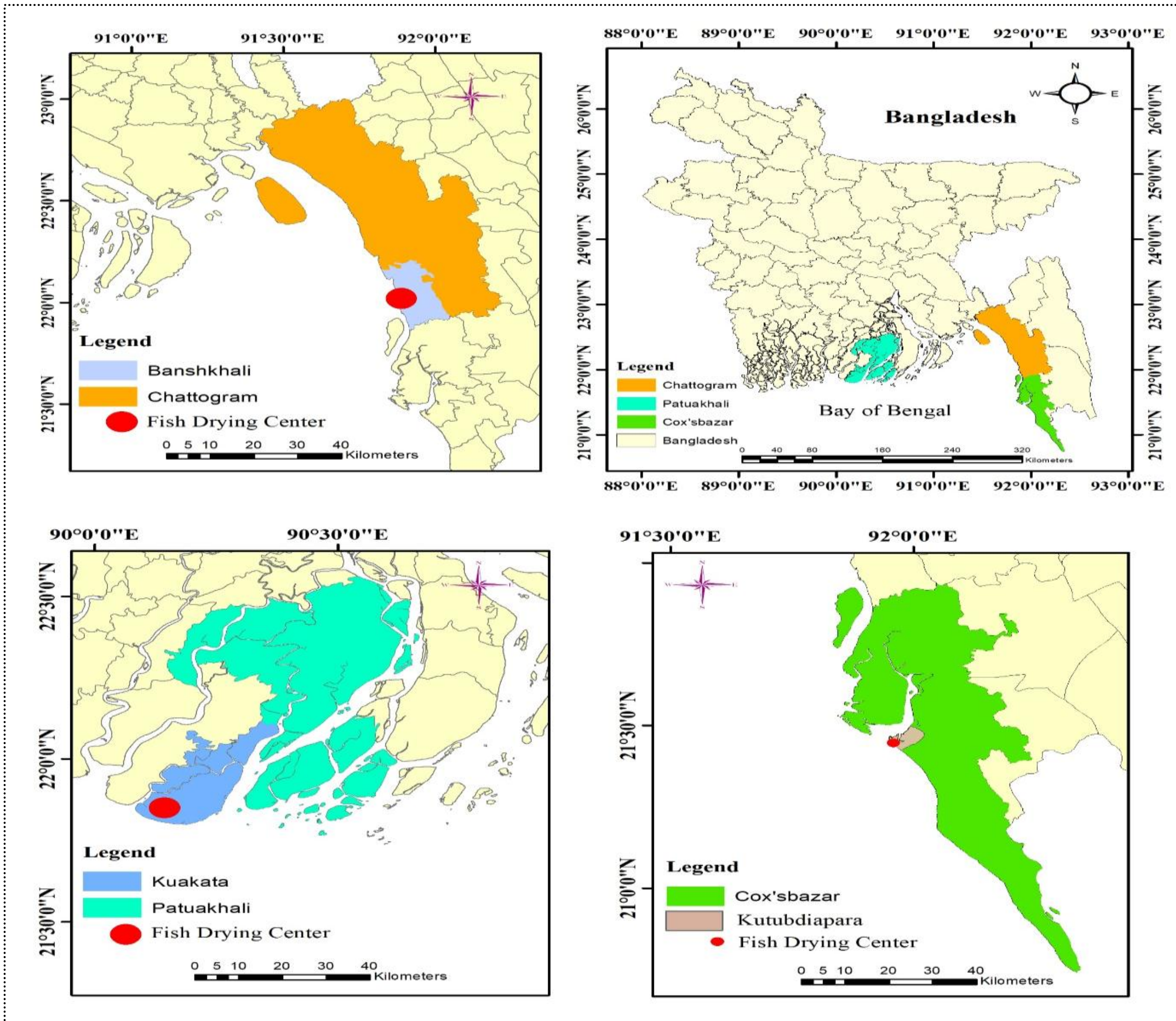

Fig. 1. Colors represent sampling stations in southern regions of Bangladesh

Microbiological analyses of sun-dried stingrays

The microbial qualities were observed by **Cappuccino and Sherman (1992)** procedure using aerobic plate count (APC) techniques, and the samples were prepared by the methodology of **ISO (1995)**. A homogeneous mixture of 1g of blended dry samples was made with 9ml of sterilized 1.5% peptone water prepared through a 1:10 dilution. Latterly, 1ml of the supernatant of the centrifuge tube was transferred, and the sample was serially diluted 10 times with 0.9% physiological saline. Aliquots of 0.1ml from each successive dilution were inoculated on the nutritional agar media for APC. For the enumeration of selective bacterial counts, 0.1ml of bacterial sample was placed on the xylose lysine deoxycholate (XLD), eosin methylene blue (EMB), *Listeria* enrichment broth (LEB) and sabouraud dextrose agar (SDA) to count the total *Escherichia coli*, total fungal count (TFC), total *Salmonella* count (TSC), total *Listeria* count and total *Vibrio* count (TViC), respectively. After 48–72hrs of incubation, colorless/ or pale pink colonies on XLD agar, purples colonies with black centers on EMB agar, fuzzy edges with white and buff on SDA agar, yellow and bluish green colonies on TCBS agar and blue-green color and an opaque halo on LEB agar were identified as *Salmonella* sp., *Escherichia coli*, fungal sp., *Vibrio cholerae* and *Vibrio parahaemolyticus* and *Listeria* sp., respectively. The following formula was used to count the above microbial communities:

$$\text{cfu/g} = \frac{\text{No.of colonies on Petridish} \times \text{Dilution factor} \times 10 \times \text{Volume of the total solution}}{\text{Weight of sample}} \times 100$$

Table 1. Characteristics count for ordaining the sensory attributes of dried scaly stingray fishery products

Organoleptic characteristics	Description	Score	Comment on quality
Color	Ash and shiny color	1-2.99	Excellent
	Slightly brownish/whitish/yellowish	3-5.99	Average
	Brownish/Faded	6-7.99	Moderately unacceptable
	Blackish color	8-10	Highly unacceptable
Odor	Natural dried fishy odor	1-2.99	Excellent
	Slight decrease of dry fish odor	3–5.99	Good
	Slightly rancid	6–7.99	Average
	Prominence of herbal odor/absence of dry fish/rancid	8–10	Poor in quality and unacceptable
Texture	Firm and flexible	1–2.99	Excellent
	Some loss of firmness and elasticity	3–5.99	Average
	Soft in texture	6–7.99	Poor in quality and unacceptable
	Brittle/Fragmented	8–10	Unacceptable
Insect infestation	No infestation	1–2.99	Excellent
	Few insects infestation	3–5.99	Average
	Moderate insect infestation	6–7.99	Poor in quality and unacceptable
	Heavy insect infestation	8–10	Unacceptable

Determination of proximate compositions

The biochemical compositions of dried stingray fishery products were determined by the methodology of standard **AOAC (2005)**. According to the procedure, moisture content was assessed by hot air oven (HAS/50/TDIG/SS, Genlab, UK) at 105°C until the constant weight (g) was achieved. The ash (A) content was obtained by muffle furnace (HM-9MP, Raypa, Spain) at 550°C for 20hrs. The kjeldahl apparatus (Bloc Digest 12, JP

Selecta, Spain) was used to estimate crude protein (CP) and a conversion factor (6.25) was used to convert total nitrogen into crude protein. The lipid/fat (F) content was evaluated by soxhlet apparatus (J-SH3, JISICO, Korea) where acetone as a prime reagent. Total carbohydrates (C) were assessed by eliminating the sum of % fat (F), % CP and % ash contents (A) from 100 **(Onyeike *et al.*, 2000)**. The total volatile base-nitrogen (TVB-N) and trimethyl amine-nitrogen (TMA-N) contents (mg N/100 g) of dried rays fish products were determined by **Antonacopoulos and Vyncke (1989)**, where a semi-automatic distilling apparatus was used to distill the digested sample after which it was titrated using a 0.01 N H_2SO_4 solution mixing indicator for ammonia titrations. For the determination of pH, firstly 10ml of distilled water was mixed with 10g of samples and then homogenized in a homogenizer (Sonic Ruptor 400, OMIN, UK) for approximately 3 min at 8000 rpm. The electrode of the pH meter (HI5522-01 Benchtop, Romania) was inserted to the prepared samples to determine the pH levels. Triplicate samples (n=3) were taken for each parameter. To calculate the caloric values, the proportions of CP, F, and C contents were combined by their respective energy values of 4, 9, and 4 kcal per 100 g of samples obtained **(Onyeike *et al.*, 2000)** using the following equation:

Caloric value = (4CP + 9F + 4C) kcal/ 100g

Determination of pesticide residues in dried stingray samples

Pesticide residues (DDT and heptachlor) were measured in dried rays samples collected from the major fish drying yards in Bangladesh.

Chemicals and reagents

About 98-99% purity (Certified Reference Materials) for DDT and heptachlor were obtained from Dr. Ehrenstorfer GmbH (Augsburg, Germany). Sigma-Aldrich Chemie GmbH (Taufkirchen, Germany) supplied acetonitrile, sodium chloride, primary and secondary amine (PSA) bond silica and anhydrous magnesium sulphate (AMS).

Preparation of pesticide standard solution

Standard stock solutions of DDT and heptachlor were made individually in acetone with a conc. of 1000mg/ l and stored at -20°C. A 50ml volumetric flask containing the accurate volumes of each stock solution and acetone was used to make an intermediate standard buffer acetone solution with a level of 50 mg/l. Another 50mg/l conc. was also prepared via the same ways. From the 10mg/ l conc. working standard solutions in acetone with concentrations of 0.1, 0.2, 0.5, 1.0, 2.0, 3.0, and 5.0 mg/l were prepared. The standard solutions were stored at -20°C until they were used.

Sample preparation, extraction and clean-up of pesticide residues

The extraction and sanitary procedures for pesticides analysis were followed, based on a modified QuEChERS method **(Prodhan *et al.*, 2021)**. The collected dried stingray samples were chopped and blended separately in an electric blender with microcutters (Preethi Steel Max MF-212, Preethi Kitchen Appliances Pty. Ltd., India) to obtain an isolated homogenous composite of dried samples. The homogenized samples (10 g) were mixed with 10ml of acetonitrile in a 50ml centrifuge tube followed by using a vortex for 1min and again mixed with 4g of AMS and 1g of sodium chloride. The mixture samples were centrifuged at 5000 rpm for 5min, and the supernatant was removed through sanitation. The supernatant (3 ml) was converted into another centrifuge containing 600 mg of AMS and 120 mg of PSA. After the centrifuge, 1ml of supernatant was filtered by a 0.2 μm PTFE filter and then taken in a vial for injection.

Operating condition of GC–MS

The samples were quantified using a Shimadzu gas chromatograph with a mass selective detector (GC–MS QP 2010 Ultra, Japan) and the analytical column, Restek (Bellefonte, PA) Rxi-5 MS with fused silica (0.25 mm internal diameter × 30 m long × 1.0 μm flim thickness). The operating mode was cleave; the cleave ratio was 10:0; the temperature (injection port) was 250°C; the sampling time was 1min; helium (carrier gas) with a flow rate of 0.75 ml/min; linear velocity was flow of control mode, 124.6 kPa pressure, 19.5 ml/min total flow, 1.5 ml/min column flow, 46 cm/s linear velocity, 3 ml/min purge flow, and the injection volume was 1μl. The column oven temperature was set and raised from 120°C to 200°C at 45°C for 3min then to 240°C at 5°C for 10min, and finally to 310°C at 10°C for 3min, where the total running time was 34min. The sample was carried out in selected ion monitoring (SIM) mode to detect selective pesticides, with a minimum of four ions considered for each pesticide. The samples were calibrated (retention time, peak area) against a five-point calibration curve of the pesticides matrix-matched calibration standard. The retention time (each peak) was used to identify through the software generated and expressed in mg/kg. The pesticides residues (DDT and heptachlor) were determined by the coefficient of determination (r^2) in the matrix-matched calibration curve (0.99), where the limits of detection (LoD) ranged from 0.002 to 0.004mg/ kg, and the limits of quantification (LoQ) were 0.01mg/ kg.

Statistical analysis

The SPSS software version 26.0 (SPSS Inc., Chicago, Illinois, USA) was used to perform for all analyses. All data were given as mean ± SE (standard error), and the least significant difference method (LSD, ANOVA) was used to analyze the differences between means. For multiple comparisons, a Games-Howell nonparametric post hoc analysis method was also applied. A value of $P<0.05$ was used to determine whether two values were significantly different.

RESULTS AND DISCUSSION

Organoleptic characteristics of dried stingray fishery products

Table (2) compares the sensorial attributes of three major fish drying yard dried stingray (*H. walga*) fishery products. There were no significant ($P>0.05$) differences among the attributes, except for the textural properties of the collected samples and the quality conditions that remained excellent for human consumption. The color of the dried rays (score=2.70 to 2.30) were bright and natural shiny, indicating that these products were of high quality, particularly for human consumption. Based on the odor score (1.60 to 1.40), there was no significant ($P>0.05$) difference among the collected products since Kuakata dried processors were produced in good condition. According to panelist, the texture of the dried ray fishery products was also noted as firm and flexible (score=1.70 to 1.30). From the insect infestation point of view, all the collected rays samples didn't differ significantly ($P>0.05$) and were perceived of high to medium quality (score=6.00 to 5.67) for human consumption. Although the overall dried rays products differed significantly ($P<0.05$) among the major fish drying yards, recommendations for acceptable and good quality still exist.

Table 2. Organoleptic characteristics of dried scaly stingray (*H. walga)* fish products

Parameter	Kutubdiapara	Chattogram	Kuakata	*P*- value
Color	$2.70^{a}\pm0.10$	$2.30^{a}\pm0.15$	$2.30^{a}\pm0.06$	0.068
Odor	$1.60^{a}\pm0.06$	$1.40^{a}\pm0.15$	$1.40^{a}\pm0.10$	0.394
Texture	$1.90^{a}\pm0.06$	$1.30^{b}\pm0.10$	$1.70^{ab}\pm0.20$	0.048
Insect infestation	$5.83^{a}\pm0.44$	$5.67^{a}\pm0.44$	$6.00^{a}\pm0.29$	0.842
Overall	$2.67^{bc}\pm0.07$	$2.85^{b}\pm0.12$	$3.09^{a}\pm0.07$	0.046

Note: Values are mean ± SE of triplicate (n= 3) samples. In a row, values with different alphabetical superscripts differ significantly at $P<0.05$.

The organoleptic assessment, however significantly ($P<0.05$) remained of good to moderate quality which is remarkably safe for human consumption. The current findings is consistent with the **Chakma *et al.* (2022)** stated that, all the sun-dried shark samples had significantly ($P<0.05$) been accepted for human consumption and expressed as excellent (score= 2.33 to 2.00). Nevertheless, **Paul *et al.* (2018)** focused on the sensory characteristics of dried stingrays products and classified them as moderately to low acceptable quality. On the contrary, the current findings are in conflict with the possibility of non-enzymatic browning reactions in dried fish, caused by ongoing color changes **(Koizumi *et al.,* 1959)**. It has been suggested that, the acceptance rate of dried fishery products would increase if the sensory score is lower and vice versa **(Roy, 2014)**. According to the current research, fishermen generated high-quality dried fisheries goods, which are in contrast to the findings of **Rasul *et al.* (2018)**, **Rahman *et al.* (2012)** and **Chakma *et al.* (2022)**.

Microbiological quality of sun-dried stingray fishery products

Table (3) summarizes the microbiological findings of traditional dried stingrays (*H. walga*) fishery products from three major fish drying locations. The APC of stingray products varied significantly (P<0.05) among the three major fish drying locations. The highest ($1.2\pm0.01\times10^6$ cfu/g) bacterial load was found in Kuakata drying yard, and the lowest ($1.2\pm0.04\times10^5$ cfu/g) was observed in Chottogram drying yard. A considerable amount of bacterial load ($1.3\pm0.03\times10^5$ cfu/g) was also found in Kutubdiapara drying yard. Likewise, the total fungal count of all dried stingray fishery product varied significantly (P<0.05) among the different location samples. The highest total fungal load ($1.5\pm0.02\times10^3$ cfu/g) was found in Kuakata drying yard and the lowest ($1.09\pm0.02\times10^3$ cfu/g) was observed in Kutubdiapara drying yard. *Coliforms* spp. and *E. coli* were also observed to be in negligible amount but exceeded the international regulatory limit in Kuakata drying yard, and the values were 21 MPN/g & 21 MPN/g, respectively. In addition, both in Chattogram and Kutubdiapara drying yard samples, the coliforms spp. and *E. coli* were also found to be less than <0.3 MPN/g & <0.3 MPN/g, respectively (Table 3). On the contrary, other pathogenic bacteria such as *Vibrio cholerae*, *Vibrio parahaemolyticus*, *Salmonella* spp. and *Listeria monocytogens* were not detected in all the collected dried stingray fish products.

Table 3. Microbial quality of scaly stingray fishery product from three major drying yards in Bangladesh

Parameter	Kutubdiapara	Chattogram	Kuakata	*P*-value
APC	$1.3\pm0.03(\times10^5)^b$	$1.2\pm0.04(\times10^5)^c$	$1.2\pm0.01(\times10^6)^a$	<0.0001
TFC	$1.09\pm0.02(\times10^3)^c$	$1.2\pm0.01(\times10^3)^b$	$1.5\pm0.02(\times10^3)^a$	<0.0001
Coliforms spp.	<0.3	<0.3	21	
E. coli	<0.3	<0.3	21	
Vibrio cholerae	ND*	ND*	ND*	
Vibrio parahaemolyticus	ND*	ND*	ND*	
Salmonella spp.	ND*	ND*	ND*	
Listeria monocytogens	ND*	ND*	ND*	

Note: ND*= Not detected; APC= Aerobic plate count; TFC= Total fungal count; Values are mean ± SE of triplicate (n = 3) samples. In a row, values with different alphabetical superscripts differ significantly at P<0.05.

The present findings of bacterial load were of higher than the regulatory limitations of **ISO, (1995)**. **Mithun *et al.* (2021)** found that, the APC of traditionally produced sun-dried fishery products may be higher (7.72×10^7 cfu/g) than that of improved dried fishery products (4.32×10^4 cfu/g). Besides, **Chakma *et al.* (2022)** stated that, the bacterial load exceeded the regulatory limits (1×10^5cfu/g) which are consistent with the present findings. The viable plate count of bacteria in dried shark can be kept low through curing the fish to the optimal moisture content and water interaction **(Al-Ghabshi *et al.*, 2012)**. This is consistent with the findings of **Nur *et al.* (2020)** who collected the fresh and dried fishes from Dhaka, Bangladesh and remarkably noted that the fish under study remained as harmful microorganisms. Unlike the current study, the most prevalent dried fishes, such as bombay duck and ribbon fish have low microbial loads that persist below the acceptable limits **(Paul *et al.*, 2018)**. The fungal count of all collected dried stingray products was reasonable, with a statistically significant (P<0.05) difference among the drying yards. The present findings revealed that the total fungal count exceeded the regulatory limits (4 log cfu/g) which is fairly consistent with the previous findings **(Feroz *et al.*, 2016; Chakma *et al.*, 2022)**. Fish are favored by fungus growth and toxin production in hot, humid climates with moisture levels over 16% and insect damage **(Hamblin, 2000)**. The harmful microorganisms, coliforms spp. and *E. coli* were found to be fewer than those recorded in the findings of **Chakma *et al.* (2022)**. Additionally, the collected dried stingray samples from fish drying yards recorded no total *S.* spp. (TSC), *V. cholera* and *V. parahaemolyticus* and *L. monocytogens*. **Chakma *et al.* (2022)** observed no colonies had grown on the respective selective media. Albeit, **Banna *et al.* (2022)** postulated that, more *Vibrio* spp. (3.7×10^5 cfu/g) were present in the dried samples, which is in contrast to the current findings.

The International Commission for Microbiological Specifications for Food (ICMSF) recommends that the standard range of food products on petri plates should be less than 5×10^5 cfu/g & moderately acceptable up to 10^7 cfu/g, while values more than 10^7 cfu/g are regarded as rejectable for human consumption **(ICMSF, 1986)**. However, there is still scope for enhancement in the quality of these delectable products by utilizing high-quality raw ingredients to make them safe food products for human consumption **(Azam *et al.*, 2003a)**. Thus, more emphasis is needed to minimize the microbial issues in dried fish from diverse locations by maintaining high-quality raw materials and enhancing procedures, sanitary and hygienic practices.

Biochemical compositions of dried stingray fishery products

The biochemical constituents of dried stingray products collected from three major fish drying yards in southern Bangladesh are shown in Table (4). All the dried stingray samples varied significantly (P<0.05), except ash and pH content. The drying yard with the highest protein content (54.78 g/100 g) was in Kutubdiapara, whereas the lowest protein content (53.72 g/100 g) was found in Chattogram drying yard. The protein content of dried ray products was also remarkably containing 54.06g/ 100g in Kuakata drying yard. In the case of lipid content, there were highly significant (P<0.05) differences among all the samples collected from major drying yards. The highest and lowest lipid values were 12.86g/ 100g in Kutubdiapara drying yard and 12.26g/ 100g in Chattogram yard, respectively. The ash content ranged from 10.98 to 10.84g/ 100g, with no significant (P>0.05) differences between the samples collected. The moisture content of all stingrays samples differed significantly (P<0.05), with the highest and lowest observed in the Chattogram fish drying yard and the Kutubdiapara drying yard, respectively. The amount of carbohydrates did not significantly (P>0.05) differ among the sampling regions, with the highest value recorded in Chattogram drying yard (1.59±0.14) and the lowest value observed in Kutubdiapara (1.20±0.13) drying yard (Table 4). For the caloric terms, significant (P<0.05) differences were detected among the collected samples. The highest value was obtained in Kutubdiapara (3.40±0.00) drying yard, and the lowest value was found in Chattogram (3.32±0.01) drying one (Table 4).

Table 4. Proximate compositions (g/ 100g) of dried scaly stingray fish products at different fish drying yards

Parameter	Kutubdiapara	Chattogram	Kuakata	*P*- value
Protein	54.78^{a}±0.16	53.72^{c}±0.15	54.06^{bc}±0.09	0.004
Lipid	12.86^{a}±0.04	12.26^{b}±0.06	12.13^{bc}±0.03	0.000
Ash	10.98^{a}±0.06	10.84^{a}±0.11	10.87^{a}±0.11	0.573
Moisture	20.18^{c}±0.07	21.59^{a}±0.23	21.55^{ab}±0.13	0.001
Carbohydrate	1.20^{c}±0.13	1.59^{a}±0.14	1.39^{ab}±0.30	0.455
Energy (Kcal/100) g	3.40^{a}±0.00	3.32^{b}±0.01	3.31^{b}±0.01	0.000

Note: Values are mean ± SE of triplicate (n = 3) samples. In a row, values with different alphabetical superscripts differ significantly at P<0.05.

The freshness indicators such as TVB-N was found in a negligible amount of quantity, and significant (P<0.05) differences were detected in all collected samples (Fig. 2). The highest and lowest quantity of TVB-N was 67.48mg N/ 100g and 62.11mg N/ 100g in Kutubdiapara drying and Kuakata yards, respectively. Notably, the stingrays samples from the Chattogram drying yard were recorded as 64.56mg N/ 100g. Likewise, the value of TMAN for all collected stingrays samples varied significantly (P<0.05), and the highest and lowest values were 49.13mg N/ 100g and 45.94mg N/ 100g in Kutubdiapara drying yard and Kuakata one, respectively (Fig. 2). The current study found that the pH value for all collected samples did not significantly (P>0.05) differ among drying yards, with the highest value found in Kutubdiapara (7.62) fish drying yard and the lowest value observed in Chattogram (7.59) yard one (Fig. 2).

Fish meat contains a variety of ingredients, including moisture, protein, lipids, vitamins and minerals, all of which contribute to the overall constituents of the fish muscle. The major composition makes up 96-98% of the total muscle constituents in fish species **(Alam, 2007)**. The protein content of dried stingray products was found to be consistent with the findings of **Azam *et al.* (2003b)**, who assessed biochemical analysis of dried stingray (*H. walga*) fish products. Albeit, some other studies conducted on the protein content of the other most popular dried fishes compatible with the present study **(Chakma *et al.*, 2022; Siddique and Aktar, 2011)**. The protein content of fish was noted to vary depending on species based on factors such as seasonal fluctuations, the effect of migration patterns and spawning seasons, food sources **(Effiong and Tafa, 2005)**. The lipid quantity of dried stingray had statistically significant difference ($p<0.05$) from all collected samples which is inconsistent with the finding of **Azam *et al.* (2003b)**, who conducted on the lipid content of dried stingray (25.3 g/100 g) fish products. The ash content in all dried collected samples were no differed significantly ($p>0.05$) but moisture content differed significantly ($p<0.05$) from one location to another drying yards. **Azam *et al.* (2003b)** investigated that the ash content and moisture content (11.01 g/100 g and 21.08 g/100 g, respectively) were consistent with the current research findings. Fresh fish has content high moisture content; drying eliminates the water and reduces the water activity, allowing the water inaccessible to pathogens and extending the shelf life of dried items which may appear low moisture in dried muscle **(Alam, 2007)**. The proximate result indicated that variability in fish species seems to possess significant influence on nutritional disparity in dried fish than geographic distinctions. Furthermore, nutritional differences from different regions may be attributed to the differences in raw ingredients sourcing, handling and processing, humidity due to processing techniques, and hygiene and sanitation practices during drying. Based on the nutritional quality, the dried fish evaluated in the present study had a higher nutritious value in terms of minerals (ash), protein and fat and excessive intake could provide safer dietary food to the country **(Siddhnath *et al.*, 2020)**.

TVB-N is a protein reduction material formed by raw fish microorganisms that is prevalently utilized as a measure of quality. Owing to the fact that such volatile bases may be generated during drying, this feature was employed to assess the rancidity of the food materials **(Reza *et al.*, 2009)**. In most practical cases, the quantity of TVB-N are 100-200 mg N/100 g widely considered as the maximum limit beyond which salt and dried fish can be recognized spoiled **(Alam, 2007)**. The present findings showed that the TVB-N and TMA-N values were higher than those prescribed by **Azam *et al.* (2003b)**, who conducted on the biochemical assessment of 14 dried species in Bangladesh. **Chakma *et al.* (2022)** revealed that a considerable amount of TVB-N and TMA-N were in acceptable range which is high compatible with the present study. However, slight variations in TVB-N and TMA-N complex in fishes were found to vary by intra to inter-species, fishing period, location, maturity level, and gender **(Sadok *et al.*, 1996)**. In most cases, the pH concentration in dried fish is maintained within allowable levels for human food. The formation of basic chemicals like ammonia and TMA is associated with a rise in pH **(Hebard *et al.*, 1982)**. The present result (7.62 to 7.59) indicated that the pH in dried stingray products were lower than the result (7.63) of **Azam *et al.* (2003b)** (Fig. 2). Changes in fish muscle pH are greatly influenced by types of fish and other physiological functions **(Chakma *et al.*, 2020)**. Moreover, the pH content is not only crucial but can also be regarded as the quality metric of a fish **(Ruiz-Capillas and Moral, 2001)**.

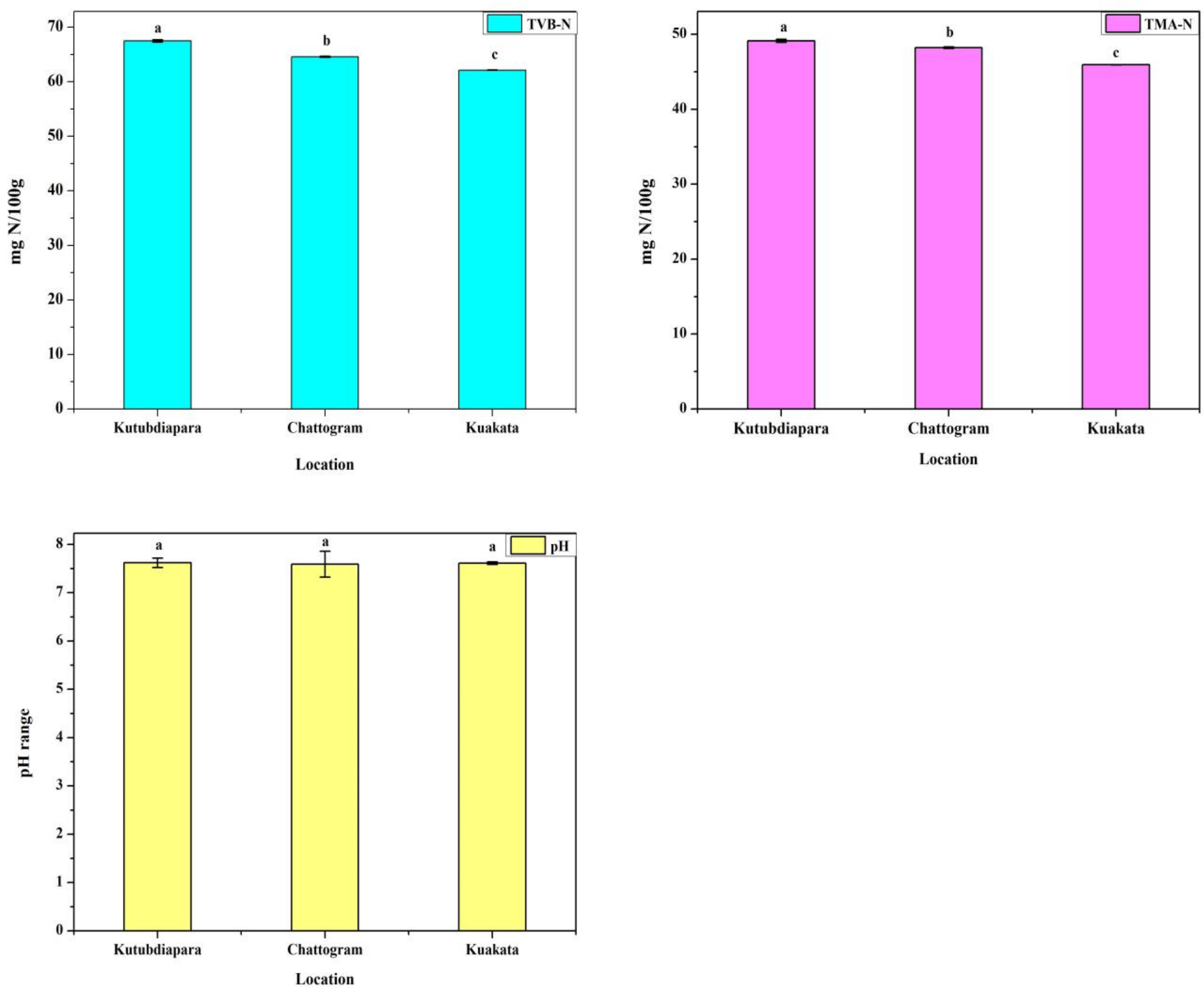

Fig. 2. Variations of spoilage indicators (TVB-N, TMA-N and p^H) dried scaly stingray fish products at different fish drying yards. Values are mean ± SE of triplicate (n = 3) samples and the values with different alphabetical superscripts differ significantly at $p<0.05$.

Pesticides contents of shark and rays dried fishery products

The prescribed pesticides were measured in dried stingray samples (n=9) collected from major fish drying yards in southern Bangladesh. According to the findings (Table 5), no dried rays samples existed pesticides (DDT and heptachlor), which are inevitably harmful to human risks. Considering that DDT and heptachlor were not detected in any of the dried samples collected from the sampling areas, there is no risk that comes with DDT and heptachlor residues in Bangladesh coastal peoples.

Table 5: Pesticide contents of dried scaly stingray fishery products from major drying yards in Bangladesh.

Samples	Pesticides	Kutubdiapara	Chattogram	Kuakata
Stingray	DDT	ND*	ND*	ND*
	Heptachlor	ND*	ND*	ND*

Note: ND* = Not Detected. Values are mean ± SE of triplicate (n = 3) samples. In a row, values with different alphabetical superscripts differ significantly at $p<0.05$.

Several studies found various pesticide residues, whereas DDT, aldrin, etc. are being commonly observed in the majority of dried fish in drying yards in Bangladesh **(Hoque *et al.*, 2021; Rasul *et al.*, 2020; Begum *et al.*, 2017; Bhuiyan *et al.*, 2009)**. Organochlorines like, DDT and heptachlor were predominantly observed in dried fish **(Sarker *et al.*, 2021)**. These results are highly inconsistent with the present findings. Despite the fact that **Chakma *et al.* (2022)** investigated that no pesticides were found on dried shark fishery products collected from southern Bangladesh. However, the variations in outputs between this research and many researchers might be ascribed by variations in collected samples, sample size, locations, and seasonal effects **(Siddique and Akter, 2011)**.

CONCLUSION

The present study examined the proximate compositions, and quality and safety aspects of dried stingray (*H. walga*) fish collected in major fish drying yards in southern Bangladesh. There were no significant (0<0.05) differences in the organoleptic properties of the collected dried products and still safe for human consumption. Despite the fact that the microbial loads of sun dried shark products surpassed permissible standards, no harmful pathogens were observed in the studied samples. In terms of biochemical compositions, dried scaly stingray (*H. walga*) fish products have higher nutritional value or quality. Furthermore, no pesticide residues (DDT and Heptachlor) were detected, and dried fish are still being posed no health risk in all collected analyzed samples. It is evident that dried stingray fishery products are good in terms of nutrients, reliability, and safety. These dried products may create valuable fishery export products and promote public health awareness both locally and internationally.

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Declaration of Competing Interest

The authors declare no conflict of interest with regard to the publication of this manuscript in your highly esteemed journal as well as there are no known contending economic interests or personal relationships that could have emerged to influence the work reported in this article.

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