Effect of salting time on formaldehyde content of dried salted Indo-Pacific mackerel

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Abstract. This paper reports an analysis of formaldehyde in dried salted short mackerel (Rastrelliger brachysoma) which was intentionally added with formaldehyde compared to those that was not. The purpose was to see whether salting could cover up the illegal use of formaldehyde. Mackerel was obtained from one day fishing landed at Karangantu Fishing Port, Serang-West Java. The fish was transported in ice by road (3 hours) to the laboratory in Jakarta, and divided into two groups upon arrival. One group was soaked in 3% (w/v) formaldehyde solution for 30 minutes, while another group was not (control treatment). They were then salted in saturated brine for 12 and 24 hours at ambient temperature and sun dried subsequently to 40% moisture content or less. The formaldehyde content of raw materials was in the range of 1.4-1.7 ppm, indicating that natural formaldehyde was present in the fish. Soaking in 3% formaldehyde solution for 30 minutes significantly increased fish formaldehyde content from 1.4-1.7 to 154-157 ppm, and decreased to 42.3-58.1 after 24 and 12h salting which then dropped to 25.0-35.9 ppm after drying, respectively. Those of control showed a slight decrease after salting and increase after drying, i.e. 2.7-3.4% in the final products. This results indicate that salting could not disguise the illegal use of formaldehyde to preserve raw materials, however small amount of formaldehyde in dried salted fish could be regarded natural formaldehyde of the fish.

1. Introduction
Salting and drying of fish are the oldest preservation techniques that are still practiced widely in Indonesia. The product is very popular in all parts of the country found in different names and forms depending upon the fish species and local customary practices. Hence, dried salted fish can be considered a staple food for the Indonesian people, especially those who live in remote areas where fish is the only available animal protein source.

Basically, all fish can be made into dried and salted products, of which the process varies from place to place. Some fish are firstly eviscerated, dressed or cut and filleted before processed, but processing whole fish is not uncommon, especially for small size; some are directly dried while others are salted first and dried. Fish that are commonly processed include small pelagic and demersal fish
such as sardines, anchovies and mackerels. The species used in the present study, Indo-Pacific mackerels (*R. brachysoma* Bleeker, 1851), also known as short mackerel, is an important commercial small pelagic fish species in Southeast Asian seas. It has a high preference among consumers because of its affordable price and widely available throughout the year.

Formaldehyde is often found in fish in many Asian and African countries such as Malaysia, India, Bangladesh, Indonesia, Vietnam, Sri Lanka, China, Tanzania and Ghana [1, 2, 3, 4, 5, 6, 7, 8]. The International Agency for Research on Cancer (IARC) classified formaldehyde as a human carcinogen based on studies of nasopharyngeal cancer and leukemia [9], and it has been prohibited for use as food additive in Indonesia [10].

There are reports of natural formaldehyde in fish available in the literature [11, 12, 13], and this formaldehyde is resulted from the decomposition of trimethylamine-N-oxide (TMAO) The use of formaldehyde to preserve fish in Southeast Asia, recorded as early as 1996 [14], is primarily due to its antimicrobial properties. For fresh fish, it is applied after catching or harvesting, either by spraying or immersing. Efforts to investigate methods to distinguish additional formaldehyde from natural ones are ongoing, but so far have not shown significant progress [15]. When reacting with muscle protein, formaldehyde will cause muscle toughness, and maintain the freshness of fish [11].

Dried fish have been reported to contain formaldehyde [16, 17, 18], and this formaldehyde can come from the natural formaldehyde of the fish, or from fish that has been preserved with formaldehyde or added during processing [19]. In Jakarta, fish for salting and drying are usually of low quality or to which formaldehyde has been added. The current paper reports on how the salting process can reduce formaldehyde, and to see if salting can mask the illegal use of formaldehyde.

2. Materials and Methods

2.1. Materials

The study was conducted during March-June 2021. The fish studied in this study was short mackerel (*Rastrelliger brachysoma* Bleeker, 1851) obtained from Karangantu Fishing Port, Serang, which was caught during one-day fishing. The size of the fish was 16-17 cm in length and 83-88 g in weight. Upon landing, the fish was transported in ice (fish to ice ratio was 1:1) to the laboratory of the Jakarta Technical University of Fisheries, which took about 3h by road. The salt used here was solar salt, while all chemicals were of technical grade. The formaldehyde stock was of 37% and diluted as necessary.

2.2. Methods

Upon arrival at the laboratory, the fish was divided into two lots. One lot was immersed in 3% (w/v) formaldehyde solution for 30 min, while the other was not. They were then salted in saturated brine for 12 and 24 h at ambient temperature. The start of salting time was set as such that both lots finished at the same time. The salted fish were subsequently sundried to 40% moisture content or less to meet the Indonesian National Standard (INS or SNI) for dried fish [20]. The experiment was conducted in three replicates. Figure 1 described at a glance the experiments.

Analysis were conducted in certain stages of the experiment as shown in tabel 1. All analysis were performed based on the methods of SNI [21, 22, 23, 24, 25, 26], except for salt content which was taken from Sudarmadji et al [27], while protein content was according to AOAC [28].
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Figure 1. Dried salted fish process in the study.

Table 1. Chemical and sensory analysis performed in the study.

| Analysis      | Raw material | After formaldehyde immersion | After salting | After drying | Refs |
|---------------|--------------|------------------------------|---------------|--------------|------|
| Protein       | ✓            | −                            | −             | −            | [27] |
| Fat           | ✓            | −                            | −             | −            | [21] |
| Ash           | ✓            | −                            | −             | −            | [20] |
| Sensory       | ✓            | −                            | −             | −            | [23] |
| Moisture      | ✓            | ✓                            | ✓             | ✓            | [24] |
| TVB           | ✓            | ✓                            | ✓             | ✓            | [22] |
| Salt          | –            | ✓                            | ✓             | ✓            | [26] |
| Formaldehyde  | ✓            | ✓                            | ✓             | ✓            | [25] |

3. Results and Discussion

3.1. Raw material composition and quality
The proximate composition, quality (such as TVB-N) and sensory scores of raw materials are presented in table 2. Mackerel is widely distributed in South and Southeast Asia, so fish landed in different countries may have the same chemical composition as shown in table 2. The mackerel in this
study was of high quality as indicated by the TVB-N and sensory scores. Fish that have a TVB-N of less than 20 or 25 mgN% are considered fresh and fit for human consumption [29, 30].

Table 2. Proximate composition and quality of raw material.

| Composition/test (unit) | Proportion/score | This study | Purwastien et al [31] | Mohanty and Nayak [32] |
|------------------------|------------------|------------|-----------------------|------------------------|
| Moisture (%wb)         |                  | 78.47 ± 0.11 | 72.9                  | 72.9 ± 0.79            |
| Protein (%wb)          |                  | 17.78 ± 0.01 | 21.1                  | 21.5 ± 0.05            |
| Fat (%wb)              |                  | 3.48 ± 0.11  | 3.8                   | 3.1 ± 0.63             |
| Carbohydrate (%wb)     |                  | 0.27 ± 0.08  | nr                    | nr                     |
| Ash (%wb)              |                  | 1.17 ± 0.15  | 1.3                   | 1.3 ± 0.37             |
| TVB-N (mgN%)           |                  | 7.79 ± 0.79  | nr                    | nr                     |
| Formaldehyde (ppm)     |                  | 1.54 ± 0.03  | nr                    | nr                     |
| Sensory (9-scale)      |                  | 8.42 ± 0.32  | nr                    | nr                     |

Table 2 also shows that fresh mackerels in this study contains natural formaldehyde, 1.54 ppm on average. This figure is not much different from that found in Noordiana [2] which reported the presence of natural formaldehyde in some fresh marine fish obtained from three different wet markets. One of them is mackerel (Rastrelliger sp) with formalin levels ranging from 1.12-1.74 mg/kg. Similarly, a figure of 1.24 mg/kg has been reported elsewhere for mackerel [33].

3.2. Formaldehyde and TVB-N content changes during processing

In this study, a group of fish were immersed in a formaldehyde solution to simulate the practice of processing dried salted fish using raw materials that have been preserved with formaldehyde. Soaking in the formaldehyde solution is also to ensure that the fish contains high enough formaldehyde so that changes during processing can be followed because the natural formaldehyde of the fish may be so small that the changes cannot be observed.

After immersion in 3% formaldehyde solution, the formaldehyde of mackerel raised about 100%, from 1.5 to 155 ppm (table 3). The formaldehyde content of mackerel then markedly dropped by 62.8% and 72.8% after 12h and 24h respectively as compared to that before salting. Formaldehyde of untreated fish slightly decreased after 12h and 24h salting, ie only by 7-20%. Sun drying further reduced the formaldehyde content of the fish by 77-84% as compared to those before salting.

The results above indicate that the longer the salting time, the greater the decrease in formalin levels in fish, and is in line with previous study [34] which reported a decrease in formalin levels in dried squid after soaking in salt solution. Soaking time of 15, 30, 60, and 90 minutes, has reduced the formaldehyde content of squid by 37.3; 49.4; 54.8 and 72.1% respectively.
| Analy (unit) | Raw material |        |        |        |        |        |        |        |        |        |        |        |        |        |        |        |        |        |
|-------------|--------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
|             |              | After salting | After drying |        | After salting | After drying |        |        |        |        |        |        |        |        |        |        |        |        |
| Moisture    | 78.5 ± 1     | 68.2 ± 1.3 | 66.1 ± 2.1 | 37.8 ± 1.3 | 38.3 ± 0.4 | 76.3 ± 0.6 | 68.2 ± 1.3 | 64.3 ± 0.2 | 37.5 ± 0.6 | ±0.3 |
| TVB (mgN%)  | 7.8 ± 1      | 13.0 ± 0.2 | 11.2 ± 0.2 | 28.6 ± 0.3 | 21.9 ± 0.4 | 8.4 ± 0.1 | 11.5 ± 0.3 | 0.7 ± 0.1 | 0.1 ± 0.3 | ±0.5 |
| Formaldehyde (ppm) | 1.5 ± 1 | 1.2 ± 0.1 | 1.4 ± 0.0 | 2.7 ± 0.1 | 3.4 ± 0.1 | 155.5 ± 1.5 | 58.1 ± 0.5 | 2.3 ± 0.5 | 8 ± 0.6 | ±0.6 |
| Salt (%)    | na           | 6.8 ± 0.4 | 8.6 ± 0.4 | 14.6 ± 0.3 | 19.3 ± 0.2 | na      | 10.5 ± 0.2 | 3.5 ± 0.1 | 4.1 ± 0.4 | ±0.2 |

na = not analysed
The increase in TVB-N observed for all fish in the present study (table 3) indicates that salting and drying cannot completely inhibit the spoilage process which in this case was demonstrated by the development of TVB-N. TVB-N is a combination of the total amount of ammonia (NH3), dimethylamine (DMA) and trimethylamine (TMA) in fish which has been widely used as an estimate of fish spoilage [35]. However, as depicted in table 3, formaldehyde has been able to add a further retarding effect of TVB-N development. The table shows that the fish that had been added with formaldehyde produced lower TVB-N for all salting times used when compared to the untreated fish.

The remaining amount formaldehyde in the final products shows that salting could not thoroughly wash out the formaldehyde from the fish, even from those that were untreated with formaldehyde. Thus if formaldehyde detected in any dried salted fish in the market, we cannot be sure whether it is originated from the fish (natural formaldehyde) or from external one. This result warrants further studies to set a maximum formaldehyde value that can be considered of fish origins. An approach to develop a relationship pattern between TVB-N and formaldehyde contents of can also be considered.

4. Conclusions
The results showed that the quality of the Indo-Pacific mackerel landed at the Karangantu Fishery Port, Serang was classified as high, which was indicated by low TVB-N and high sensory characteristics. Formaldehyde in the process of salting and drying is able to slow down the development of TVB-N. Salting for 12-24 hours was not able to completely reduce the formaldehyde content in fish. This means that salting cannot cover up the use of fish that has been added with formaldehyde. Further research is needed to determine the value of formaldehyde content in dried salted fish which can be ascribed to the decomposition of TMAO.

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