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RESEARCH PAPER

Effect of Season and Chilled Storage On Biochemical Composition of Tunisian Caramote Shrimp Head and Common Cuttlefish Viscera

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Abstract

Seasonal changes of biochemical composition of caramote shrimp head (CSH) and common cuttlefish viscera (CCV) were investigated. The effect of chilled storage (5°C) of these by-products during 21 days on the physicochemical quality indicators was evaluated for every season to assess a suitable storage period. All biochemical components varied significantly ($P<0.05$) overall season for both types of by-products except moisture and chitin contents, which remained constant in CSH (79.77±0.33% and 8.33±1.11% respectively). CCV and CSH by-products were a main source of proteins (28.37±5.44% and 20.37±5.77% respectively on dry weight basis (d.w.b)) and ash (23.60±4.18% and 28.28±2.32% respectively d.w.b). Principal component analyses (PCA) proved that CSH could be upgraded to obtain a rich product in minerals and chitin components at any time regardless the season. CCV collected in spring and summer could be assimilated as a homogenous group rich in protein and lipid. The chilled storage CCV and CSH during 21 days showed significant ($P<0.05$) variations of some physicochemical indicators and according to PCA, by-products might be stored just for few days (not more than a week) as proteolysis and lipolysis increased from the first week.

Keywords: Seafood by-products, components characterization, refrigeration, quality indicators.

Introduction

Fishing has been an important socioeconomic activity for a long time on Tunisian coasts. It produced about 119000 tones and created employment for roughly 54 miles persons (DGPA, 2014). Among seafood products, shellfish (Parapenaeus longirostris, Penaeus kerathurus, Metapenaeus monoceros, Aristeus antennatus) and cephalopods (Sepia officinalis, Octopus vulgaris, Eledone moschata and Loligo vulgaris) were extremely researched by the majority of processing industries to be exported to European markets. A great part of these products were distributed to freezing's industries where they were processed and inedible fractions were eliminated. During shrimp processing, head, tail and shell could be removed and residues represented approximately 33–65% of the raw material weight and they depended on species and their size (Sachindra and Mahendrakar, 2005; Pacheco *et al.*, 2009). For cephalopods species, cuttlefish by-products (viscera, gonads, beak, skeleton, ink sac, skin, eyes, and gills) presented 30-35% of raw material weight (Aloulou *et al.*, 2006; Le Bihan, 2006). Several studies were conducted to look for potential uses of seafood by-products because they proved that shrimp wastes are a main source of, proteins, chitin, calcium, carotenoid and lipids with a high contents of astaxanthin / ω–3 fatty acid ratio (Sadighara *et al.*, 2014) such as docosahexaenoic acid: DHA and eicosapentaenoique acid: EPA (Babu et al., 2008; Sánchez-Camargo *et al.*, 2011). These components could be used in diverse applications including food, feeds, cosmetic, agriculture and medical industries (Park *et al.*, 2001; Nargis *et al.*, 2006; Oliveira Cavalheiro *et al.*, 2007). Cephalopods by-products were rich in proteins, amine acids, minerals and vitamins (Abdelmouleh, 1997; Sadok *et al.*, 2004). It was demonstrated in particular the interest of ink for its antibacterial activity and its use in food, cosmetic and painting industries (Saidane, 2001). Nevertheless, the industrial processing of these by-products is limited by the variability of their chemical composition and their production. (Palpandi *et al.*, 2009) proved that amount of components (humidity, ash, protein and fats) of shellfish and cephalopods by- products vary according to species and seasons (Green and Martrick, 1979; Cho *et al.*,

1998), in addition to anatomic fraction analyzed (Thirunavukkarasu and Shanmugam, 2009). Besides seafood processing and storage conditions accelerate their alteration.

In Tunisia, seafood by-products are still discarded in great quantity and then contributed to heighten environmental pollution. Because shrimp and cephalopods by-products stocks are irregular and depend extremely on several factors (annual production, processed quantity, seasonal variation of biochemical composition), up-grading of these organic matter needs to look for useful, economic and environment friendly processes. The knowledge of physicochemical composition variation of seafood by-products is a very important step before application of any processing. Furthermore, as seafood products are highly perishable due to bacterial activity, their storage at low temperature until use is necessary. Freezing at temperature inferior to -10°C stopped completely and irreversibly any metabolic activity and allowed to preserve for a long time more than a year these products (Cheftel and Cheftel, 1977; Gonçalves and Gindri Junior, 2009).

The present study is the first work dealing with biochemical characterization and variation of quality indicators during chilled storage of caramote shrimp (Penaeus kerathurus) and common cuttlefish (Sepia officinalis) by-products from Tunisian industries. Seasonal changes of biochemical components (moisture, protein, lipid, ash and chitin) of CSH and CCV throughout the year were determined. For each season, effect of storage at 5°C along 21 days on physicochemical quality indicators : pH, Soluble Nitrogen (SN), Total Volatile basic nitrogen (TVB-N), Thiobartituric Acid Reactive Substance (TBARs) and Acid Value (AV) were determined. The by-products were examined weekly in order to decide the suitable storage period, ensuring minimal degradation of the principal biochemical components of CSH and CCV.

Materials and Methods

Raw Material Samples

P. Kerathurus head (CSH) and S. officinalis viscera (CCV) were collected from a Tunisian freezing industry (Sfax, Tunisia) within the 24 hours following products processing. These species were caught in the gulf of Gabes (Tunisia). By-products were transported immediately in icebox to the laboratory. A total quantity of each by-products (1.25 kg i.e. ~30% of raw P. Kerathurus and ~24% of raw S. officinalis) was separated in five aliquots and kept frozen (-25°C) until analysis. 250 g of each by-product was conserved to biochemical composition and the four aliquots of 250g each one were conserved to evaluate physicochemical quality indicators during storage at 5°C. Mixed-sex of CSH, corresponding to cephalothoraxes (heads) were sampled on July, November and April (2011-2012). Mixed-sex of CCV and consisting of gonads, ink sac, hepatopancreas, gills, eyes and skins were gathered on July, October, February and May (2011-2012).

Chemicals

Hydrochloric acid (HCl, Purity 37%, analytical degree) and concentrated sulfuric acid (H2SO4, 98%, analytical degree) were supplied by Panreac Quimica S.L.U®. Butylated Hydroxy Toluene (BHT), potassium hydroxide (KOH) and sodium hydroxide (NaOH); were obtained from Labo Chemie®. Trichloroacetic acid (TCA, 20%) was provided by Carl Roth®. The 2-thiobarbituric acid (TBA) and chloroform (CHCl3), were obtained from Sigma-Aldrich®. Nitric acid (HNO3, 65%) was purchased from Carlo Erba® and ethanol (C2H6O) from Scharlab S. L®. All the solvents used were of analytical-grade.

Biochemical Composition

Raw material samples were analyzed according to AFNOR (2001) procedures. All analyses were determined in triplicate during every season: autumn, winter, spring and summer. Each sample of CSH and CCV were ground in a blender (Moulinex 400W) and then dried at 47°C until obtained 12-13% of residual moisture. Dried matter was homogenized in flour for protein, lipid and chitin analyses. Moisture was quantified by oven-drying at 105°C to constant weight. Ash was obtained by incinerating the dried residue for 4 hours at 550°C in a muffle furnace (Schaltplan Modell, NABERTHERM Brand, and KARL KOLB Home). Total nitrogen (N) was estimated according to Kjeldahl (1883) method and protein was estimated as Nitrogen corrected x 6.25. Correction consisted of subtraction of total nitrogen from chitin's nitrogen. The lipid contents were analyzed according to Soxhlet (1879) lipid extraction method by using chloroform at 60°C for 6 hours, followed by removal of the solvent under vacuum at 40°C. Chitin contents were estimated according to Welinder (1994) method. Determination of chitin is based on demineralization followed by deproteinization, drying and incineration. Demineralization was carried out by using a solution of Ethylenediaminetetraacetic acid (EDTA: 1 mol l-1) instead of using of hydrochloric acid solution (1 mol l-1). Deproteinization was performed by using a solution of NaOH (2 mol l-1).

Determination of Physicochemical Quality Indicators

In order to evaluate physicochemical quality indicators of CSH and CCV collected after a series of industry processing steps, an aliquot of each by-product was taken and grounded with a blender

(Moulinex® 400W). Fresh matter was used for pH, TVB-N, SN, AV and TBARs. pH value was measured according to AOAC (2000) with the use of pH-meter (Mettler Toledo GmH, MP220). Stage of advanced spoilage of CSH and CCV was evaluated by TVB-N analyses according to the regulation CE N°2074/2005. Values were expressed as mg of N/100 g of sample. Autolysis reaction and degree of hydrolysis of protein into other components such as amino acids and short chain peptides was estimated by determination of SN according to the method described by Lo, Liao, Bullock and Jones (1993). Lipid hydrolysis was determined by acid value (AOCS, 1997) after fat extraction according to Folch, Lees and Sloane-Stanley (1957) method. Lipid oxidation was evaluated by the 2-thiobarbituric acid method (TBARs) as described by Vyncke (1975). The result is expressed in mg malonaldialdehyde/kg sample.

Statistical Analysis

Results are given as the mean values ± standard deviation for triplicate analysis. All data were analyzed using the STATISTICA 8.55 Software (1984-2004; StatSoft, Inc., Tulsa, OK, USA). Samples of CSH and CCV collected at every season were considered independent. Biochemical components (moisture, protein, lipid, ash and chitin) and physicochemical quality indicators (pH, TVB-N, SN, AV and TBARs) were the dependant variables. Seasonal variation of biochemical components was analyzed using ANOVA one-way. Previously, normality and variance homogeneity were checked by Shapiro-Wilk test and Levene test, respectively. When data did not obey to ANOVA condition, the non-parametric ANOVA equivalent (Kruskal–Wallis test) was performed. Significant differences were revealed by Tukey HSD test and multiple comparison of mean rank Test, respectively. To compare the nutritional quality of shrimp and cuttlefish by-products, mean values of biochemical components were compared using T-Student test (or non-parametric test Mann-Whitney U).

Effect of storage at every season on physicochemical quality indicators for each type of seafood processing by-products was evaluated using ANOVA tow-ways factors with replications (paired samples). Differences were considered statistically significant when $P<0.05$. Significance level of all tests applied was fixed at 95%. In order to decide to upgrade samples of each by-product regardless the season and storage period, principal component analysis (PCA) was performed on the correlation matrix of the biochemical variables (PCA1) and on the correlation matrix of physicochemical indicators variables (PCA2). A measure of association between each original variable and the obtained principal components was provided.

Results

Seasonal Composition of Caramote Shrimp and Common Cuttlefish By-Products

The proximate composition of caramote shrimp head (CSH) and common cuttlefish viscera (CCV) determined in summer, autumn, winter and spring is shown in Figure 1.

Moisture

P. kerathurus and S. officinalis by-products showed elevated moisture content, with a mean content of 79.77±0.33% and 79.76±1.44% respectively. No significant difference ($P>0.05$) in moisture value during seasons was recorded for CSH, although CCV demonstrated seasonal significant variation ($P=0.04$) with maximal value recorded in autumn (82.02±0.11%). Comparison of moisture content between CCV and CSH showed no significant difference ($P=0.00$) according to Student test.

Ash

Ash mean content varied from 23.60±4.18% (CCV) to 28.28±2.32% (CSH), with a significant seasonal difference ($P<0.05$) observed for both by-products (Anova and Kruskal–Wallis test). Concerning CCV, maximal value of 29.14±0.10% was observed in winter and minimal value (18.04±0.88 %) was noticed in summer. According to Student test, CSH was significantly richer in ash than CCV.

Protein

CCV were characterized by an average protein value equal to 28.37±5.44% against 20.37±5.77% for CSH, with significant difference ($P<0.05$) throughout the year for both by-products. The HSD Tukey test showed that higher value of protein was recorded in summer CCV. For CSH, protein content didn't differ from summer and autumn season. Thus, mean value of protein for CCV was significantly higher than protein content for CSH (Student test).

Lipid

P. kerathurus and S. officinalis by-products were a lower fatty product according to Ackman (1989) classification as mean percents of lipid were respectively 9.84±2.52% and 10.29±8.56%. Seasonal means of lipid for both by-products varied significantly ($P<0.05$) and significantly higher values of lipid percentage were observed in spring for CCV. Student test proved that CSH and CCV had no significant difference ($P=0.10$) on lipid content.

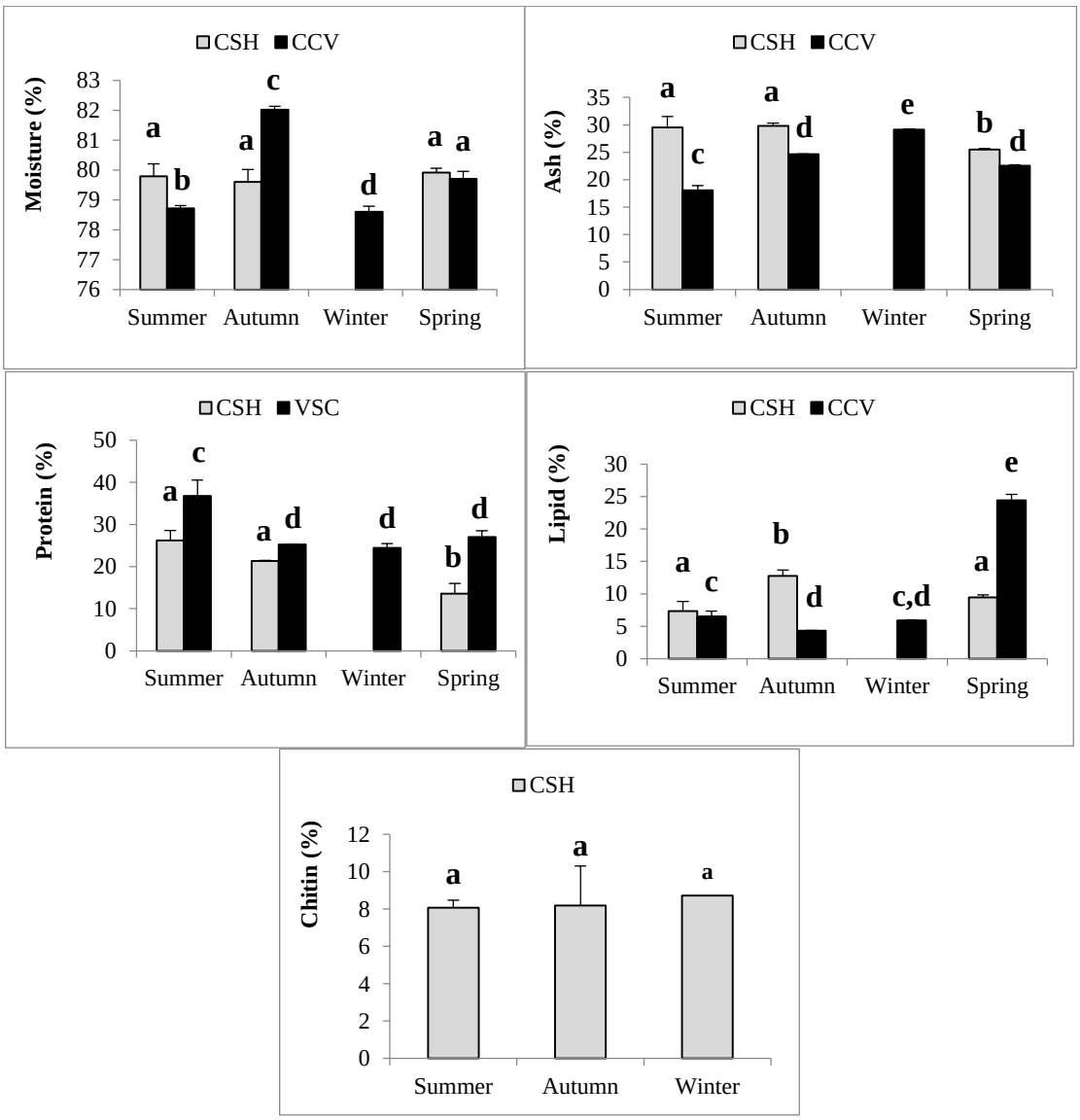


Figure 1. Seasonal variation in moisture (% wet weight basis) ash, protein, lipid and chitin (% dry weight basis) contents in caramote shrimp head (CSH) and common cuttlefish viscera (CCV). Vertical rectangles of histograms with different letters are significantly different (P<0.05).

Chitin

Chitin is a naturally abundant polysaccharide after cellulose (Das and Ganeds, 2010). Chitin contents in CSH were constant during the year and the average value was 8.33±1.11%.

Principal Component Analysis and Seasonal Biochemical Components of Shrimp and Cuttlefish By-Products (PCA1)

The relationship between biochemical components (moisture, protein, lipid, ash and chitin) determined at every season were investigated by principal component analysis (PCA1). Results of PCA1 revealed that 85% of the variation among the seasonal samples of shrimp and cuttlefish by-products was attributed to the first three principal components (Table 1 and Table 2). The first principal component (PC1) explained 44% of the variance. It associated protein, chitin and ash contents (Figure 2). Protein content was positively opposed to the group of ash and chitin values presented in the negative side of PC1. Regarding the relationship between seasonal samples of CSH and CCV and principal components, PC1 associated CCV, determined in summer and spring (in positive side), to CSH samples determined in summer, autumn and spring (Figure 2). Thus, it could be concluded that PC1 showed that CCV are rich in protein (in summer and spring respectively) than CSH, which presented high value of chitin and ash (summer, autumn and spring, respectively). The second principal component (PC2) explained 22% of the variance and it was defined by the lipid and moisture contents which are also opposed. The observation of the distribution of by-products samples

Table 1. Eigenvalues of correlation matrix using CSH and CCV and five variables (mean data- PCA1)

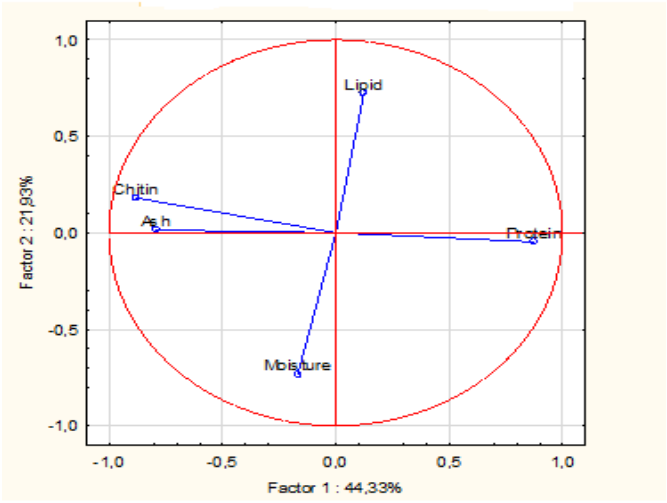
Value number	Eigenvalue	% Total of variance	Cumulative eigenvalue	Cumulative
1	2	44.33	2.22	44.33
2	1	21.93	3.3	66.26
3	1	19.18	4.27	85.44
4	0	9.61	4.75	95.05
5	0	4.95	5.00	100.00

Table 2. Eigenvectors of correlation matrix using CSH and CCV and five variables (mean data, PCA1)

Variable	Factor 1*	Factor 2**	Factor 3	Factor 4
Moisture	-0.11	-0.7	0.65	-0.13
Protein	0.58	-0.04	-0.24	-0.34
Lipid	0.083	0.69	0.67	-0.23
Ash	-0.53	0.01	-0.23	-0.81
Chitin	-0.59	0.17	-0.07	0.38

*Factor1: Protein, chitin and ash
**Factor 2: Lipid and moisture

A



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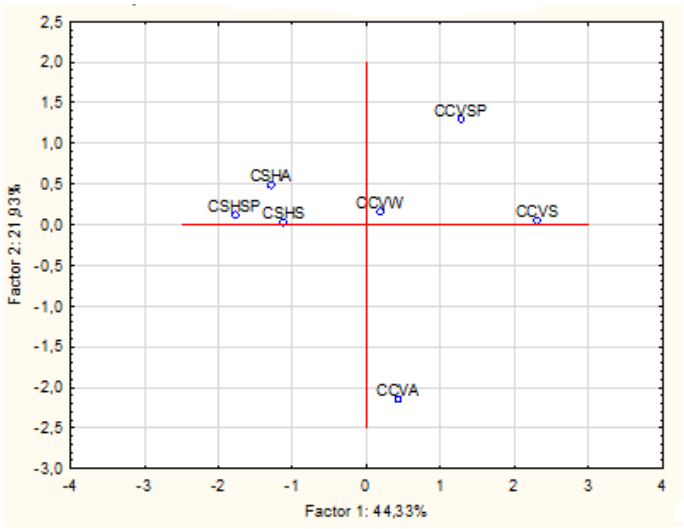


Figure 2. Projection of the variables (**A**) and shrimp and cuttlefish samples (**B**) on the factor plane Factor1: Protein, chitin and ash; Factor 2: Lipid and moisture (PCA1) CSHS: Caramote shrimp head summer; CSHA: Caramote shrimp head autumn; CSHSP: Caramote shrimp head spring; CCVS: Common cuttlefish viscera summer; CCVA: Common cuttlefish viscera autumn; CCVSP: Common cuttlefish viscera spring; CCVW: Common cuttlefish viscera winter.

highest value of lipid content inversely to autumnal samples much richer on moisture content.

The third principal component (PC3) contributed by 19% to the variability and it was represented also by lipid and moisture contents. For the other principal components, having an eigenvalue less than 1.0 and explaining a small percentage of the variance, were not considered.

Seasonal Evolution of Physicochemical Quality Indicators During Chilled Storage

Changes in physicochemical quality indicators (pH, TVB-N; SN, FFA and TBARS) of shrimp and cuttlefish by-products (CSH and CCV) were investigated during chilled storage (5°C) for 21 days at every season.

pH

Averages pH values of CSH and CCV determined before chilled storage were 8.29±0.13 and 7.53±0.21 respectively. A significant ($P<0.05$) decrease of pH was observed during chilled storage at every season (Figure 3) followed by a significant increase ($P<0.05$). Although the significant fall of pH, CSH kept basic character and CCV became acid at the 14th days in autumn and winter then neutrality of viscera was observed. Fluctuation on pH mean values was significantly ($P<0.05$) observed after a week of storage.

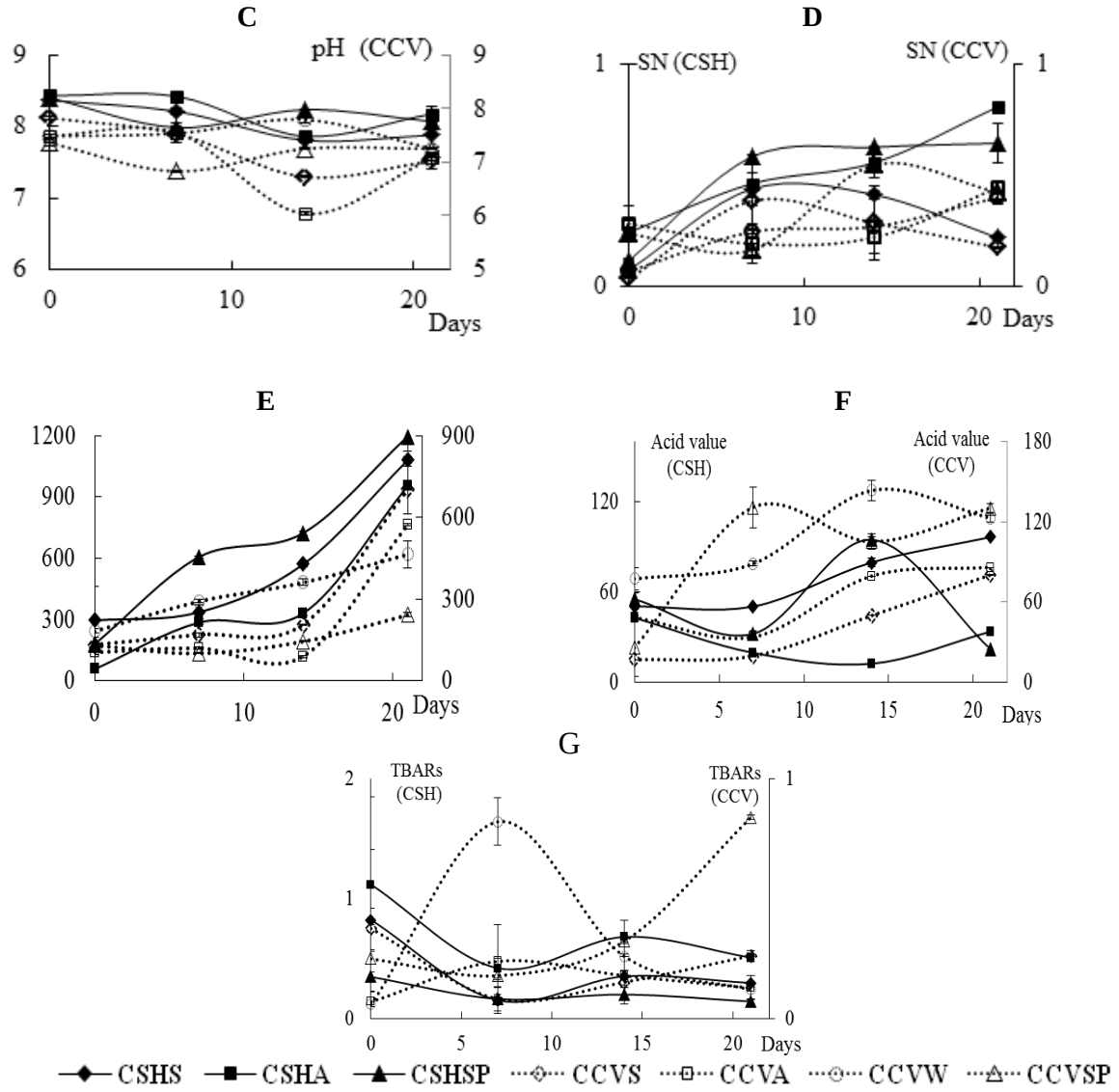


Figure 3. Evolution of pH (C), soluble nitrogen (g N/100 g w.w.b, D), total volatile basic nitrogen (mg N/100 g w.w.b, E), Acid value (mg KOH/g, F) and Thiobartituric Acid Reactive substance (mg MDA/kg) mean values of shrimp and cuttlefish by-products during chilled storage. CSHS: Caramote shrimp head summer; CSHA: Caramote shrimp head autumn; CSHSP: Caramote shrimp head spring; CCVS: Common cuttlefish viscera summer; CCVA: Common cuttlefish viscera autumn; CCVSP: Common cuttlefish viscera spring; CCVW: Common cuttlefish viscera winter.

Soluble Nitrogen (SN)

SN resulting from the protein decomposition varied significantly (P<0.05) during chilled storage respectively for CSH and CCV (Figure 3) at all seasons. Maximal values of SN were recorded at 21th days of storage for CSH in autumn (85% of total nitrogen) and spring (73% of total nitrogen) seasons. For CCV, the high value of SN was observed in spring (60% of total nitrogen) at the 14th day. Thus, proteolysis activity was more pronounced in CSH than CCV. Student test showed that seasonal mean values of SN for both by-products differed significantly (P<0.05) in exception values taken at day 0 for autumnal samples and at 14th day for summer samples.

Total Volatile Basic Nitrogen (TVB-N)

Determination of TVB-N (mg/100g of sample) showed a significant increase (P<0.05) during chilled storage in all seasons. Maximal value was recorded in 21th day for both CSH and CCV (Figure 3). Comparison of TVB-N determined in both by-products revealed seasonal significant differences (P<0.05) for the whole period of storage.

Acid Value (AV)

During chilled storage, seasonal mean values of acid index varied significantly (P<0.05) for CSH and CCV (Figure 3). According to Anova test, mean values of acid index of CSH (49.39±27.13mg/KOH) were slightly superior to acid index of CCV (42.61±0.00mg/KOH) with maximal value observed in winter (77.57±5.85mg/KOH) for CCV.

Thiobartituric Acid Reactive Substance (TBARs)

Both CCV and CSH showed low initial values of TBARs with the exception of the autumnal value for CSH which was a little superior (Figure 3). This result could be in accordance with the fact that higher lipid content gives the higher values in fish samples (Tokur *et al.*, 2006) and as mentioned above, P. kerathurus and S. officinalis by-products were a lower fatty product. During chilled storage, means values of TBARs showed no seasonal significant difference (P>0.05) for CCV and maximal value was recorded at the 21th day in spring (0.84±0.01meq of MDA/kg). However, seasonal mean values of TBARs of CSH varied significantly (P<0.05) during storage.

Principal Component Analysis and Seasonal Physicochemical Quality Indicators of Shrimp and Cuttlefish By-Products (PCA2)

Principal component analysis (PCA2) was applied to physicochemical quality indicators (pH, SN, TVB-N, AV and TBARs) which measured the spoilage evolution of CSH and CCV during chilled storage. The results are presented in Table 3 and Table 4. The first principal component (PC1) explained 36% of the variance. It was presented by SN, TVB-N and pH variables which are negatively projected. SN and TVB-N variables presented a group and pH variable another group (Figure 4). The projection of seasonal CSH and CCV determined at every storage period on the factor plan opposed the samples of CCV, analyzed in the summer (day 14), autumn (day 14), winter (day 0) and spring (day 7), to both groups composed of autumn and spring samples of CSH (storage day 7 and 14 and 21). Therefore, the

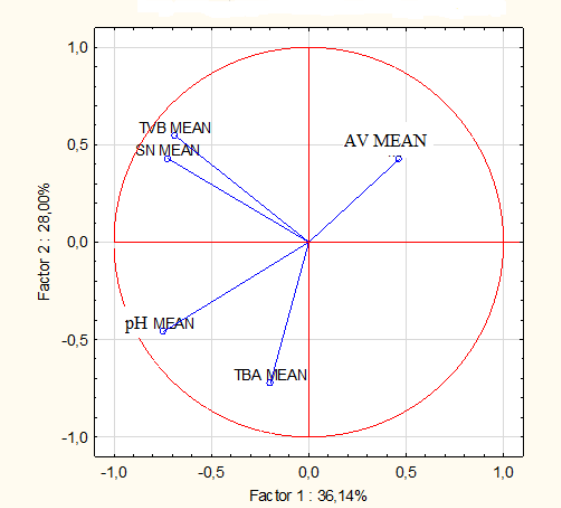
Table 3. Eigenvalues of correlation matrix using CSH and CCV and five variables (mean data; PCA2)

Value number	Eigenvalue	% Total of variance	Cumulative eigenvalue	Cumulative
1	2	36.14	1.81	36.14
2	1	28	3.21	64.13
3	1	18.47	4.13	82.61
4	1	10.67	4.66	93.28
5	0	6.72	5	100

Table 4. Eigenvectors of correlation matrix using CSH and CCV and five variables (mean data; PCA2)

Variables	Factor 1*	Factor 2**	Factor 3	Factor 4	Factor 5
Soluble nitrogen (SN)	-0,54	0,36	0,09	0,69	0,31
Total volatile basic nitrogen (TVB-N)	-0,51	0,46	0,19	-0,41	-0,57
Acid value (AV)	0,35	0,36	0,76	-0,18	0,37
Thiobartituric Acid Reactive substance (TBARs)	-0,15	-0,61	0,61	0,30	-0,38
pH	-0,56	-0,39	0,06	-0,48	0,55

*Factor1: SN, TVB-N and pH;
** Factor 2: TVB-N and TBARs



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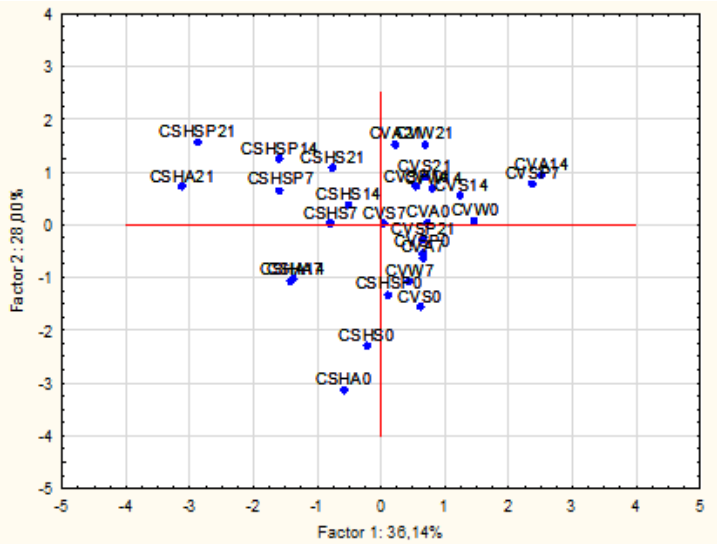

(J)

results lead to deducing that CSH collected in autumn and spring presented higher values in SN and TVB-N than CCV. The evolution of SN and TVB-N were inversely correlated to the pH value of the product. The second principal component (PC2) explained 28% of the variance and it was defined by the TVB-N variable which is opposed positively to TBARs variable (Figure 4). Samples of CCV stored during 21 days in autumn and winter presented elevated values of TVB-N than samples CSH, taken at every season (day 0), and summer samples of CCV. The third principal component (PC3) contributed by 18.5% to the variability and it was represented by TBARs and AV (Figure 4). The factor opposed samples of CCV collected in summer (storage days 0, 7), autumn (storage days 0, 7) and spring (storage days 0) to samples of CCV collected in winter (storage days 0, 7) and spring (storage days 21) and autumnal samples of shrimp (storage day 0). The result proved that lipid oxidation and hydrolysis was important in samples of CCV collected in winter and spring for the storage period ranging from a week to three weeks.

Discussion

The biochemical composition of caramote shrimp head (CSH) and common cuttlefish viscera (CCV) collected from Tunisian seafood processing industries was investigated. Obtained results could help industries decide to look for economic process to valorize this organic matter. Both CSH and CCV showed elevated moisture content and these results agreed with the fact that humidity is the major component in seafood products (up to 80%) and water activity was high (Zakhia, 2002). In comparison with other results, moisture content in CSH found in our study was similar to the value (79.1±0.8%) obtained by Heu *et al.* (2003) in the case of Pandalus borealis by-products (head, shell and tail). Although, our value was superior to the moisture average values reported by Babu *et al.* (2008) in the heads of Penaeus monondon (67.4±4.6%), Penaeus indicus (73.6±1.0%)

and Metapenaeus monoceros (75.8±0.6%). Mizani and Aminlari (2007) described a little superior value of moisture in the shrimp head (80.5±0.3%). Concerning CCV, Abdelmouleh (1997) reported that moisture content varied from 75.9% (autumn) to 78.7% (spring) and the mean value (77.2%) was inferior to mean value found in our study. In addition, Castro, Garriw and Sow (1992) and Soufi-Kechaou *et al.* (2009) obtained inferior values of moisture in S. officinalis viscera (74.4 and 77.2%). Difference in moisture amount may be explained by difference in anatomical structure of by-products as the shrimp head was supported by the shell, so water retention capacity could be different. Furthermore, time elapsed from fishing to processing steps, in addition to freezing, which affected cells contributed to loss important quantity of water (Cheftel and Cheftel, 1977).

Concerning ash content, richness of shrimp species in ash was documented by several authors and values ranged from 7.50 to 40.50% on a dry basis (Abulude *et al.*, 2006; Sánchez-Camargo *et al.*, 2011). As CSH contained shell, which was composed of organic molecules and minerals in particular calcium carbonate, that's explained higher values of ashes. Synowiecki and Al-khateeb (2000) observed similar values to our results for shrimp shell (29.2±0.2%). For CCV, ash values were more superior to the values found by Soufi-Kechaou *et al.* (2009) for viscera of the same species collected from the same fishing area (2.0% w. w. b against 4.83%). The significant difference in amount of ashes was more important for CCV than CSH and the difference could be the result of heterogeneity in nutriments (shells, fishes and skeletons) remained in the stomach.

Furthermore, CSH and CCV were a main source of protein. Several studies proved that protein represents the major component of shrimp heads (Fagbenro and Bello-Olusoji, 1997; Cira *et al.*, 2002; Randriamahatody *et al.*, 2005; Coward-Kelly *et al.*, 2006; Limam *et al.*, 2008; Cao *et al.*, 2009) and values differed according to shrimp by-products. These values varied from 13.7% to 70.3% on a dry basis (Mirzah, 1990; Okoye *et a.l*, 2005; Dey and Dora, 2011; Balogun and Akegbejo-Samsons, 1992). For CCV, significant seasonal variations of protein content were also observed by Kacem *et al.* (2011) in their study of S. officinalis viscera. Ozogul *et al.* (2008) found that S. officinalis had significant ($P<0.01$) high level in proteins in autumn than in spring and winter. This result was different to our results. In the other hand, results obtained in this study were inferior to the protein value obtained by Blanchier and Boucaud-Camou (1982) (86% on d. w. b) in viscera and by Rosa *et al.* (2005) in digestive gland of S. officinalis (49.3±3.6%). In his work, Abdelmouleh (1997) found inferior protein value in S. officinalis viscera (16.2% on d. w. b) in comparison with values obtained in our study.

The fact that CSH was a lower fatty product was confirmed by Sánchez-Camargo *et al.* (2011), who found in Brazilian redspotted shrimp (Farfantepenaeus paulensis) waste (head, shell and tail) a value of lipid content equal to 4.83 ± 0.06% d.w.b. Also, Tsape *et al.* (2010) reported that P. kerathurus fished on October in North Aegean contained fat in the proportion of 2.4±0.1% (wet weight basis) in the head. This value was near values found in this study, although shrimp was caught in November (1.9±0.7% w.w.b). Because of absence of adipose tissue in shellfish, the midgut gland or hepato-pancreas present in the head was the main storage organ of energy reserves (Chang, 1995; Garcia *et al.*, 2002). However, fat could be stored in muscle tissue and in female gonads (Komatsu and Ando, 1992; Shenker *et al.*, 1993). According to Wouters *et al.* (2001), dietary requirements of shrimp are generally higher in sexually maturing adults and during maturation. The weight of the ovaries increases four to eightfold (Mourente and Rodriguez, 1991; Ravid *et al.*, 1999). Furthermore, molt cycle influenced significantly lipid content of the midgut gland increased in inter-molt shrimp cycle (Chang, 1995). Guary *et al.* (1976) documented that lipid content of shrimp varied in amount and composition with the time of the year and these seasonal changes have been attributed to one or more of the following factors: water temperature, food, stage of development, sex and photoperiod (Bottino *et al.*, 1980). About CCV, seasonal changes may be associated with size, stage of sexual maturity and spawning period limited to spring and in some area to late June (Gauvrit *et al.*, 1997). These confirmed our results consisting that high level of lipid was recorded in spring (24.41±0.93%). Meanwhile, it could be explained by nutritive conditions (Blanchier and Boucaud-Camou, 1982; Rosa *et al.*, 2005). Blanchier and Boucaud-Camou (1982) and Castro, Garriw, Sow (1992) have found slightly higher value (12% and 16.9% on d. w. b) of lipid content in S. officinalis viscera and digestive gland respectively.

Besides protein, lipid and ash components CSH was characterized by chitin content. Ashford *et al.* (1977) observed superior value of chitin of shrimp (14-27% on d. w. b) and crab (13-15%) processing wastes, respectively. In the work of Mizani and Aminlari (2007), the chitin content of P. semisulcatus head (6% on d. b) was inferior to our value. However, Synowiecki and Al-Khateeb (2000) and Cao *et al.* (2009) found superior value (11%) on shrimp head and Crangon crangon by-products respectively. Results obtained in this study (2.1±0.1% wet basis) agreed with those obtained by Babu *et al.* (2008) who found near value (2.4±0.6% wet basis) in CSH.

Based on the principal component analysis (PCA1), season seems to be the most important factor in the fluctuation of biochemical components for both species. Furthermore, CSH could be considered as a homogenous group, regardless of the season and could be upgraded to obtain a rich product in minerals

and chitin components at any time of the year. CCV collected in spring and summer could be assimilated as another homogenous group rich in protein and lipid.

The second part of this study dealing with storage of CSH and CCV at 5°C at 21 days showed the limit of refrigeration to conserve these by-products for a long period until valorization. Changes in biochemical composition during storage were mentioned at each season for both CSH and CCV. About pH values, inferior value (pH = 6.5) was recorded by Le Bihan *et al.* (2007) in S. officinalis viscera generated by transformation factories. This value remained significantly stable during 24 hours for CCV preserved at 4°C. After 4 months of storage, pH increased progressively and reached 6.8. The observation of pH in samples mantle of S. officinalis stored on ice during 13th day, showed an increase of pH over time (Sykes *et al.*, 2009). They explained that the increase of pH might be related to the ‘‘sponge’’ effect, since the individuals accumulate water in their tissues leading to a loss of acidity. According to other authors, the increase of pH after decrease could be the result of proteolysis activity which release amine acids, volatile bases nitrogen (Jackson *et al.*, 1997; Ruiz-Capillas *et al.*, 2002) and of releasing of free amine acids due to microorganisms (Sainclivier, 1985). Concerning CSH, the profile of decreasing and increasing of pH was observed as well as by Gonçalves and Gindri Junior (2009) in frozen shrimp immerged in ice and stored at -18°C during 180 days. In the work of Jeyasekaran *et al.* (2006), the pH value of Penaeus indicus catched in May decreased from 7.9 to 7.3 during 12 hours of storage under ice. Decrease of pH during storage could be the result of the anaerobic production of acids (lactic, acetic, butyric, propionic) produced by spoilage anaerobic bacteria characteristic of marine seafood products (Huss, 1999; Qingzhu, 2003). In general, pH value depends on several factors such as time spent since fishing, temperature of storage and physiological state of the animal (Moral, 1987). Consequently, pH value changes after chilled storage and tends to exceed 7.0 due to accumulation of basic metabolites during bacterial spoilage (Hebard *et al.*, 1982).

Proteolysis activity in both CSH and CCV was significantly proved by the simultaneous fluctuation and increasing of SN and TVB-N during storage. TVB-N resulted from protein degradation due to bacterial enzymes or endogenous enzymes present in the fish. It represents the sum of ammonia (NH3), dimethylamine (DMA), trimethylamine (TMA) and others amines with low molecular weight (volatile). TVB-N is an indicator of spoilage of some fish species (red fish, flat fish, gadoids, hake and Atlantic salmon). However TVB-N cannot be used as a freshness indicator in the early stage of storage (constant level during the first days of iced storage) and does not reflect the mode of spoilage, bacterial or autolytic (Huss, 1995, Baixas-Nogueras *et al.*, 2002). In general, it reflects only stages of advanced spoilage of fish (Sykes *et al.*, 2009) and it is considered unreliable for the measurement of spoilage during the first 10 days of cod’s ice storage as well as for several other species (Huss, 1999). Values of TVB-N found in our study were higher than values found by Heu *et al.* (2003) for both species of shrimp Pandalus borealis and Trachypena curvirostris where values didn’t exceed some 10mg/100g in by-products composed of head, sell and tail. The studies mentioned the increase of TVB-N for different marine species (Trachurus mediterraneus, Sepia aculeata and Photololigo duvaucelii, Sepia officinalis and Illex illecebrosus) during storage under ice at 2±1°C ($P<0.01$) (Vaz-Pires *et al.*, 2008; Mbarki *et al.*, 2009; Tantasuttikul *et al.*, 2011). This increase could be the result of producing ammonia from bacterial catabolism of nitrogenous compounds (Okeyo *et al.*, 2009; Liu *et al.*, 2010). According to Ruiz-Capillas *et al.* (2002), main changes of this parameter were occurring when animals were stored in cool temperature. In their study, Abu Bakar *et al.* (2008), observed a rapid increase in TVB-N at 10°C due to the increase in total aerobic bacteria in fresh and preserved Macrobrachium rosenbergii. The microbial activities caused the decrease in the amino acids arginine, lysine and histidine, which correlated well with the increase in the corresponding biogenic amines such as putrescine, cadaverine and histamine respectively.

High content of SN observed after processing of marine product resulted from bacterial degradation and also the manipulation after capture in cephalopod species (Ruiz-Capillas *et al.*, 2002). According to Sykes *et al.* (2009), cephalopod spoilage is mainly autolysis. Due to their composition, protein fraction undergoes a fast degradation after death caused by both endogenous and bacterial enzymes, where proteases play a major role (Hurtado *et al.*, 1999). Lipid changes of CSH and CCV stored at 5°C during 21 days were followed by determination of AV and TBARs. When lipids were decomposed, important quantities of free fat acids were accumulated in the tissue during storage (Aubourg *et al.*, 2004). This phenomenon was more observed in CCV than CSH due to lipase present in digestive gland (Mancuso *et al.*, 2014). Values of acid index recorded in both raw materials of by-products were highly superior to values characteristic of cod oil liver (5.6mg of KOH/g oil; Speight and Lange, 2005). Lipid hydrolysis was due to lipid enzymes and this phenomenon was highly active at temperature ranging from –4 to 4°C and weak at freezing (Aubourg *et al.*, 1998).

Concerning TBARS, the 3-carbon compound malonaldehyde (MDA) is a major carbonyl decomposition product of auto-oxidized, polyunsaturated lipid materials and TBARs present an important quality indicator for fatty fish (Tokur *et al.*, 2006). The TBARs values of CCV samples determined in autumn and winter increased during the

first week of storage and decreased for the rest of the period. These variations could be the result of decomposition phases of peroxides, formation of carbonyls and interactions of components with nucleophiles molecules or use of MDA by microflora (Kasimoglu *et al.*, 2003). Studies have shown that increases in the TBARs number up to a certain point during the storage period, followed by a decrease in these values (Gomes *et al.*, 2003). During the evaluation of lipid oxidation in stored foods, decreases in TBARs values are probably due to interactions between malonaldhyde and protein (Igene and Pearson, 1979). Another reason is that during the analysis method of TBARs value, addition of 1% BHT in the sample during fat extraction method resulted in the lower value of TBARs (Salih *et al.*, 1987). Tsaknis *et al.* (1999) observed that in non-oxidized dried-salted Octopus vulgaris, MDA concentration was 0.12mol/L of MDA, and in oxidized products, this value was 2.21 mol/l of MDA. Non-oxidized product was similar to the values found in shrimp head. In non-oxidised salted fatty fish (Clupea harengus), TBARs test was 2.84±0.12 mol/L MDA and this value increased to 8.50 mol/L MDA. In our study, the raw materials stored during the 21 days at 5°C contained lower concentrations of oxidized compounds and it was concluded that storage period has no effect on oxidation of shrimp and cuttlefish by-products.

According to principal component analysis (PCA2), season and storage period contributed significantly to enhance spoilage of shrimp and cuttlefish by-products. A group of samples of CCV stored for a week presented inferior value in SN, TVB-N, pH, TBARS and AV in comparison with samples stored for more than a week (14-21 days). Concerning CSH, storage period (day 0) showed a product with a very low oxidized product even though, from the first week, high levels of decomposed nitrogenous product were detected.

Conclusion

Penaeus kerathurus head and Sepia officinalis viscera are a good source of organic (protein, fat and chitin) compounds and ash. These components varied significantly ($P<0.05$) during season and results showed the limit of fluctuation of each variable. According to principal component analyses, shrimp head could be considered as a homogenous group regardless of season and could be upgraded to obtain a rich product in minerals and chitin components at any time of the year. Cuttlefish by-products collected in spring and summer could be assimilated as another homogenous group rich in protein and lipid. The evaluation of quality indicators (pH, SN, TVB-N, AV and TBARs) of P. kerathurus and S. officinalis by-products during chilled storage at every season revealed that these by-products couldn't be stored more than a few days at 5°C (not more than a week) as proteolysis and lipid activities were elevated from the first week. Other analyses (amino-acids, fatty acids) and microbial analyses will be completed in the next investigation to provide a complete characterization of these seafood by-products which will be useful for industrial valorization.

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