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Quality Indicators of Northern Shrimp (*Pandalus borealis*) Stored under Different Cooling Conditions

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ABSTRACT

The quality changes of northern shrimp, stored in ice, liquid-ice or salt-water ice at either -1.5°C or 1.5°C, were evaluated by using sensory assessment, chemical analysis, bacteriological test and physical methods. The main objective of this study was to identify freshness and quality indicators of Northern shrimp (*Pandalus borealis*) and to evaluate the efficiency of different cooling conditions. The total volatile nitrogen (TVB-N) level in shrimp stored in liquid ice decreased during the first day of storage, and TVB-N formation was delayed at least for 3 days for shrimp in liquid ice stored at -1.5°C. In other shrimp stored in ice or salt-water ice, the TVB-N level increased with the time of storage. The trimethylamine (TMA) value increased gradually with storage time in all samples, except for the one stored in liquid ice at -1.5°C during the first day of storage. The salt content increased rapidly in shrimp stored in liquid ice at -1.5°C and increased slowly in other storage conditions, except for the iced shrimp where the salt content decreased slowly during the storage period. Water content increased gradually for all samples during storage. Texture showed only minor changes. Total viable counts (TVC) showed that bacteria grew most quickly in shrimp stored in ice and in salt-water ice, followed by those in liquid ice at 1.5°C and -1.5°C, respectively, throughout the storage period. Liquid ice storage at -1.5°C gave the longest shelf-life of shrimp based on sensory analysis. Statistical analyses, principal component analysis (PCA) and analysis of variance (ANOVA), shows good correlation between, TVB-N, TMA, TVC, pH, NH_3 response of electronic nose and sensory evaluation.

Keywords: Northern shrimp (*Pandalus borealis*); freshness; spoilage; sensory evaluation; liquid ice; superchilling; electronic nose.

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1 INTRODUCTION

The northern shrimp (*Pandalus borealis*) is primarily harvested in Newfoundland and Labrador and it is estimated that 20,000 tons were landed in 2001 (Project summary, 2002). Iceland is a major producer of cold water shrimp *(Pandalus borealis)*. From 1989 to 1997 the annual catch of this species increased from 27,000 to over 80,000 tons. Most of the shrimp is iced on board the vessels and processed in factories around the country within 5-7 days from the time of catch (Valdimarsson *et al*., 1998).

Shrimp is a perishable product. Its shelf life and wholesomeness during refrigerated storage and shipping is greatly influenced by both enzymatic and microbiological changes. Shellfish spoil more rapidly than fish for a number of reasons. Firstly, they are smaller, and small fish spoil more rapidly than larger ones. Secondly and more importantly, the gut is usually not removed immediately after capture, hence postmortem autolytic changes will occur faster. A third reason is that the chemical composition of shellfish tissue is different and it contains a lot of non-protein nitrogenous compounds that encourage more rapid spoilage (Aitken *et al*. 1982, Shamshad *et al*. 1990). Black spot, or melanosis, a discoloration indicative of spoilage always occurs in shrimp (Jeong *et al* 1991). Therefore, it is important for the shrimp processing industry to develop a storage method to maintain high quality and freshness of shrimp.

Fish and shellfish are highly perishable and the quality deterioration of raw seafood is usually dominated by microbial activity. This deterioration is highly temperature dependent and can be reduced by low storage temperature. Raw seafood deterioration has two forms: microbiological and non-microbiological. Non-microbial deteriorations, both enzymatic and non enzymatic also contribute to the spoilage changes. Micro-organisms are present on the external surfaces and in the gut and head of shrimp. Upon death, the micro-organisms or the enzymes they secrete are free to invade or diffuse into the flesh where they react with the complex mixture of natural substances present (Lee and Um1995).

Due to the perishability of such a product, freezing is often used in fisheries industry and frozen products are most common in many processing companies. However, deterioration of texture and flavour is a frequent problem for frozen products. Fresh seafood products stored in ice, including fresh shrimp, has always been the consumer's primary choice. Preservation methods for fresh shrimp have been applied to extend shelf- life and to avoid health hazards. Such methods include chilled storage in ice (Shamshad *et al*. 1990, Rogério *et al* 2001, Lakshmanan *et al*. 2002), in liquid ice (Huidobro *et al* 2002), modified ice storage (Jiang *and Lee* 1988), superchilled storage at $0^{o}C \sim -4^{o}C$ (Aleman *et al*. 1982, Fatima *et al*. 1988), modified atmospheres packaging storage in ice (Baka *et al*. 1999; Lopez-Caballero *et al*.2002), gamma radiation (Yeh and Hau 1988), and treatment with organic acids and their salts (Benner *et al*. 1994, Mosffer 1999). Liquid ice has recently been introduced as a successful method for the rapid chilling of seafood products and a way of reducing the temperature of products below those attained with traditional ice. Traditional iced storage presents some undesirable attributes, e.g. injury and bruising of the products (Huidobro *et al*. 2002).

The evaluation of quality and shelf-life of seafood is based on sensory, chemical and microbiological tests. Chemical test, for example tritmethylamine (TMA), total volatile nitrogen (TVB-N), K value and acid-TBA, etc. are commonly employed (Botta 1995, Jackson *et al*. 1997, Nielsen 1997). However, chemical methods and some physical methods need laboratory facilities and trained staff. Moreover, these methods are destructive, i.e. seafood once examined cannot then be sold.

There is little information on the quality deterioration of the shrimp stored in liquid-ice or salt-water ice at subzero temperatures, which is one of the most efficient ways of chilling storage. A comprehensive study is needed to identify freshness and quality indicators of shrimp stored at zero and subzero iced, liquid-iced and in salt-water iced storage. For this reason, quality change and shelf-life of shrimp stored under different cooling conditions using ice, liquid-ice or salt-water ice was investigated by sensory, chemical, microbiological, physical methods. It is necessary to find out a method that is practical and accurate for evaluating the freshness and quality changes of shrimp.

2 LITERATURE REVIEW

2.1 Quality deterioration of shrimp

Most important factors in raw seafood are freshness and quality. Upon death, there are pronounced changes in the appearance, texture, chemistry, and redox potential of the muscle. In postmortem muscle, the conversion of ATP to ADP, ADP to AMP, and AMP to IMP usually takes place within 24 h or less. These changes are thought to be totally autolytic since, in most instances, insufficient time has elapsed to allow the proliferation of spoilage microorganisms. Several factors can affect the rate of IMP accumulation, including temperature, species, and handling. The initial loss of the attributes characterising freshness in seafood results primarily from catabolic changes in nucleotides and carbohydrates, which are rapidly followed by degradative reactions of nitrogenous compounds as well as hydrolysis and peroxidation of lipids. These reactions are catalyzed mainly by endogenous enzymes during further chilling of the catch and bacterial activity contributes to the quality deterioration (Norman and Benjamin 2000).

It has been known for many years that both bacterial and enzymatic changes are responsible for fish spoilage. Uchiyama and Ehira (1974) reported that for cod and yellowtail tuna, enzymatic changes related to fish freshness preceded and were unrelated to changes influenced by microbial activity. In shellfish, the freshness deterioration is additionally related to enzymatic discolorations known as blackspot (Jeong *et al*. 1991). After catch, the enzyme, especially polyphenoloxidase (PPO), is responsible for the formation of melanins causing darkening of the meat and shell. These black spots occur on raw and undercooked prawns (Norman and Benjamin 2000). Reducing the activity of endogenous enzymes and preventing PPO action slows down the rate of deterioration during storage of shrimp. Various techniques and methods have been developed over the years to prevent PPO action, and to reduce the activity of endogenous enzymes in seafood. These methods and techniques include processing, utilizing heat treatment, refrigeration, freezing, dehydration, irradiation, high-pressure, and the use of browning inhibitor.

2.2 Chilling and superchilling storage of raw seafood

Chilled or iced preservation during storage, distribution and retailing are necessary to prevent browning in shrimp. This is based on the idea that refrigerated temperature is effective in reducing enzymic activity. The rate of enzyme-catalyzed reactions is controlled to a great extent by temperature. It has been found experimentally that increasing the temperature from 0^{o}C to 10^{o}C at least doubles the rate of spoilage of fish flesh and the controlling of temperature and time is of prime importance in reducing deterioration of raw material (Norman and Benjamin 2000).

Storage of fish at temperatures between 0°C and -4°C is called superchilling or partial freezing. Superchilling extends product shelf life, but a negative effect on freshness/prime quality has been observed for some fish species. The prime quality of superchilled shrimp from Pakistan was increased from 8 days in ice to 16 days in NaCl/ice at -3°C (Fatima *et al*. 1988). Also, both freshness (measured by a K-value of 20%) and shelf life of cultured carp (*Cyrinus carpio*), cultured rainbow trout (*Salmo gairdnerii*) and mackerel (*Scomber japonicus*) have been improved by superchilling at -3°C as compared to storage at 0°C (Aleman *et al*. 1982). Fresh Atlantic salmon fillets packaged under modified atmosphere (MA) were stored in superchilled (-2°C) and chilled (4°C) conditions, and the results show that superchilled salmon stored at -2°C had a 21-d sensory shelf life (Sivertsvik 2003). Lee and Toledo (1984) reported that the microscopic ice crystals formed at -2°C longitudinally between the muscle bands which kept the muscle fiber apart and could not have been rigid enough to separate muscle fibers.

Liquid ice is a new superchilling technique for food that requires less time to chill products and acts more uniformly than other types of traditional ice. Liquid ice is composed of millions of microscopic spherical ice crystals suspended in seawater or brine (Optimar, 2003). These structure characteristics provide the ice with a superior ability to chill fish due to its better heat exchange power and to prevent marking or physical damage to the fish (Huidobro *et al*. 2001). The practical advantage of liquid ice is its pumpability that it can be pumped through conventional pipes and is storable in all type of tanks or containers. Moreover, on account of the microscopic size of the ice crystals, the main benefit of liquid ice is its ability for rapid chilling of fish and to provide lower fish temperature.

2.3 Assessment methods of freshness and quality

2.3.1 Sensory evaluation

Sensory evaluation is an important method for the assessment of freshness and quality, and is commonly used in the fish sector and fish inspection services (Martinsdottir 1997, Luten and Martinsdottir 1997). Sensory evaluation can be applied to all species of fish and laboratory facilities are not necessary. The evaluation is quick and non-destructive unless the sample is being cooked, and moreover, the results often reflect the criteria the consumer uses in evaluating acceptability (Connell 1990). Therefore, when chemical and physical methods are being used for assessing the quality of fish, sensory evaluation should be conducted to ensure that the results of the instrumental (objective) tests are in agreement with sensory analysis and thus indicating consumer perception (Alasalvar *et al*. 2001). The quickest way, used by buyers and inspectors on the market, is to look at the appearance of the fish products, particularly the colour, luster of the shrimp. The disadvantages are that the evaluations of inspectors are difficult to standardize and the results can be subject to the personal whims and biases of the assessors. However, most trade is based on sensory assessments, although measurements are not always objective and documented. The Quality Index Method (QIM), which as a method of sensory evaluation, is a grading system based on adding demerit points for sensory attributes used for estimating the freshness and quality of seafood. The QIM has been demonstrated to be rapid and more objective than sensory classification schemes often used by the industry. QIM schemes have recently been developed for a number of fish species including: fresh herring, cod, red fish, Atlantic mackerel, mackerel, European sardine, brill, dab, haddock, pollock, sole, turbot, shrimp and farmed Atlantic salmon (Sveinsdottir *et al*. 2003).

2.3.2 Chemical analysis

Several chemical tests for freshness such as determination of amines, particularly trimethylamine (TMA), and determination of hypoxanthine have been used for the past decades (Aitken *et al*. 1982). The former is related to bacterial activity while the latter is a measure of enzymic change. These two methods complement each other and have different ranges of applicability and usefulness. A chemical test does not measure freshness directly but the two are associated because the concentration of chemicals measured is dependent on storage time and temperature, as freshness is.

Trimethylamine, TMA, is formed in spoiling fish by the action of certain species of bacteria on the substance trimethylamine oxide, TMAO. Therefore determination of TMA content is a measure of bacterial activity and spoilage (Aitken *et al*. 1982). Increase in TMA during iced storage is similar to the increase in bacterial numbers. TMAO is not only an important compound for maintenance of physiological functions in fish and shellfish but it is also a key substance in the spoilage of raw or processed seafood (Norman and Benjamin 2000). The TMAO content in the muscle of crustaceans is 9-28 (mmole/kg wet weight) (Konosu and Yamagushi 1982).

The measurement of total volatile basic nitrogen (TVB-N) is often used as an alternative to measuring TMA content because the TVB-N value includes mainly the content of ammonia, trimethylamine, and dimethylamine. Therefore, changes in TVB content during spoilage are very similar to those of TMA except that the initial value is much higher.

ATP degradation patterns in fish, shellfish, crustaceans, and cephalopods can be used to estimate the freshness and quality of fish (Norman and Benjamin 2000). The K or "freshness" index gives a relative freshness rating based primarily on the autolytic changes which take place during *post mortem* storage of the muscle. Thus, the higher the K value, the lower the freshness level. Hypoxanthine is the end product of a series of enzymic reactions going on in the flesh. Unlike TMA and TVB, hypoxanthine increases in most species soon after death and in the early days of storage.

The development of TMA is in many fish species parallel to the production of hypoxanthine. Hypoxanthine can be formed by the autolytic decomposition of nucleotides, but it can also be formed by bacteria; and the rate of bacterial formation is higher than the autolytic. Both Jorgensen *et al*. (1988) and Dalgaard *et al.* (1993) showed a linear correlation between the contents of TMA and hypoxanthine during iced storage of packed cod. None of these chemical indicators that include total base nitrogen (TVB-N), biogenic amines, trimethylamine (TMA), dimethylamine (DMA), K value, etc., however, is universally applicable (Gill 1990, Botta 1995).

2.3.3 Microbiological methods

The activity of microorganism is the main factor limiting the shelf life of raw seafood. Microorganisms are found on all the outer surfaces (skin and gills) and in the intestines of live and newly caught fish. The total number of organisms vary enormously and Liston (1980) states a normal range of 10^2-10^7 cfu (colony forming units)/cm^2 on the skin surface. The gills and the intestines both contain between 10^3 and 10^9 cfu/g.

When the fish dies, the immune system collapses and bacteria are allowed to proliferate freely. On the skin surface, the bacteria to a large extent colonize the scale pockets. During storage, they invade the flesh by moving between the muscle fibres. Murray and Shewan (1979) found that only a very limited number of bacteria invaded the flesh during iced storage.

An estimation of the total viable counts (TVC) is usually used as an acceptability index in standards, guidelines and specifications (Olafsdottir *et al*.1997c).

2.3.4 Physical measurements

Chemical methods have some operational disadvantages such as being destructive, requiring some laboratory facilities and taking a long time to complete. Therefore, new methods are needed that will measure rapidly properties of fish related to freshness and display the result simply.

Electronic nose measurements: Recently, electronic noses have been introduced as alternative rapid techniques to supplement or replace traditional quality control techniques in the food industry. Electronic nose systems have been designed to be used for quality control of raw and manufactured products; process, freshness and maturity monitoring; shelf-life investigation; microbial pathogen detection, etc. (Schaller *et al*. 1998). The electronic nose is promising for application in food industries where rapid measurements with no sample preparation are needed to detect microbial spoilage (Olafsdottir *et al*. 2002). An electronic nose FreshSense based on electrochemical gas sensors (CO, SO_2 and NH_3) has been used for freshness monitoring of various species of fish i.e. haddock, capelin, redfish and cod (Olafsdottir and Jonsdottir 2003). The sensitivity of the sensors towards different compounds is different, for example, CO sensor has high response to the production of alcohols and NH3 can detect the formation of amines etc. (Olafsdottir *et al*. 2002). The responses of the electrochemical sensors correlate well with classical methods to evaluate freshness and spoilage of seafood, i.e. TVB measurements and sensory analysis, for capelin (Olafsdottir *et al*. 1997a, 2000) herring and fresh roe (Olafsdottir *et al*. 1997b), and whole or peeled shrimp (Högnadottir, 1999).

Texture measurements: Some characteristics in shrimp that result in the decline of freshness and quality are mainly related to structure, appearance (including colour), odour, water-holding capacity, etc. Texture is a very important property of fish product whether it is raw or cooked. Texture measurement can be used to determine structural changes. The four principal quality factors in food are the appearance (comprising colour, shape, size, gloss), flavour (comprising taste and odour), texture, and nutrition (Malcoim 2002). Texture of raw fish can be measured by different methods using mechanical food testing equipment. The main techniques applied for fish are puncture, compression, shear, and tensile stress. Among them, the shearing force and compression methods are recommended for use with fresh fish (Sigurgisladottir *et al*. 1999). When the texture of raw fish is measured, hardness and springiness are often the major variables (Botta 1991). Hardness was defined as the maximum force during the first compression cycle (first bite) and has often been substituted by the term firmness. Its units are N (force). Resilience is a measurement of how the sample recovers from deformation both in terms of speed and force derived. It is taken as the ratio of areas from the first probe reversal point to the crossing of the x-axis and the areas produced from the first compression cycle. It is not a parameter from the original Texture Profile Analysis (TPA) work but instead has been developed from looking more closely at the elastic recovery of the sample. Springiness (originally called elasticity) is related to the height that the food recovers during the time that elapses between the end of the first bite and the start of the second bite. There is no unit for this parameter. Cohesiveness is defined as the ratio of the positive force area during the second compression to that during the first compression. Tensile strength is a manifestation of cohesiveness. This parameter is unitless.

Figure 1 shows a typical TPA curve generated by the G. F. Texturometer. The height of the force peak on the first compression cycle (first bite) was defined as hardness (Malcoim 2002). In Figure 1, A is the beginning of the first compression and B is the beginning of the second compression. The ratio of the positive force areas under the first and second compressions (A_2/A_1) defines cohesiveness. The distance that the sample

recovered its height during the time that elapsed between the end of the first bite and the start of the second bite (BC) was defined as springiness (originally called elasticity).

Texture profile analysis (TPA) is an objective method of sensory analysis pioneered by Szczesniak (1963), who defined the texture parameters first used in this method of analysis. Later, Bourne (1978) adapted the Instron to perform TPA by compressing standard-sized samples of food twice. TPA is based on the recognition of texture as a multi-parameter attribute. For research purpose, a texture profile in terms of several parameters determined on a small homogenous sample is desirable.

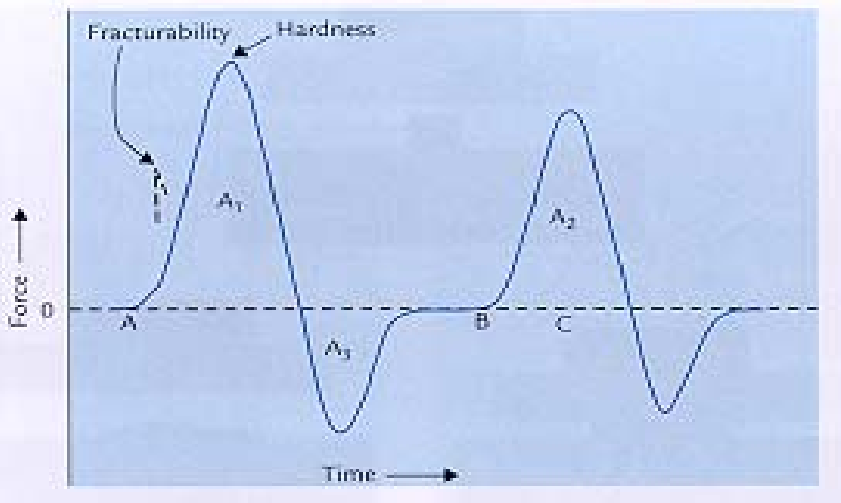

Figure 1: A typical texture profile analysis (TPA) curve (Malcolm 2002).

3 MATERIAL AND METHODS

3.1 Raw material and experimental design

Northern shrimp (*Pandalus borealis*), was caught in Arnarfjordur (Westfjords, Iceland) on the 2nd of December 2003 and stored in isothermic boxes containing crushed ice, followed by truck transport to the IFL laboratories in Reykjavik two days after catch. The temperature of shrimp was 4.5^{o}C and the ice had melted in the boxes holding the shrimp when they arrived at the laboratory.

The shrimp was randomly divided into 4 groups that were kept under different conditions. The groups were stored in ice at 1.5^{o}C of ambient temperature (room temperature) (ICE/+), in liquid-ice at 1.5^{o}C (LIQ/+), in liquid-ice at -1.5^{o}C (LIQ/-) and in salt-water ice at -1.5^{o}C (S-ICE/-), respectively (Table 2). A thin layer of liquid ice and ice were put in the bottom of the bin, then a layer of shrimp, about 5 cm height, was laid into the bin and covered with liquid ice and ice again. This was repeated until the bin was filled up (the layers of shrimp kept in each bin were five). All the bins were covered by liquid-ice, ice, or salt-water ice on top and kept at temperature of 1.5^{o}C (ICE/+ and LIQ/+ groups) or -1.5^{o}C (S-ICE/- and LIQ/- groups). The centre temperature of every storage bin was

measured with 1 h intervals using automatic record-meter inserted in the four bins. Temperature of the cold chamber was also monitored. The liquid ice was supplied by Optimar (Company, in Iceland) with initial salt content of 3.5% and ice content of 27%~30%. The flack ice was made of potable water at the laboratory.

The ratio of ice to shrimp had been theoretically calculated taking into account how much ice was needed to chill the shrimp down from 4-5°C to 0°C and how much ice to keep the shrimp chilled for 10 days (Table 1). The mass of ice used (M_i used) was 5-6 times the calculated minimum value the mass (M_i total) determined theoretically. This was done to make sure that there would be enough ice for the during the whole storage period.

Table 1: Latent heat of fusion (ΔH_f) of the different cooling agent, the minimum quantity needed to cool the shrimp and keep it chilled (Mi total) and the quantity used for 15 kg of shrimp (Mi used).

Type of cooling agent	Ms * Cps * ΔT (kg)*(kcal/kg°C)*(°C)	Ratio of ice (%)	ΔHf (kcal/kg)	Mc for chilling (kg)	Mc for storage (kg)	Mi total (kg)	Mi used (kg)
Flake ice	15*0,8*10=120	100	80	1,5	2,3	3,8	**15,0**
Liquid ice	15*0,8*10=120	30	24	5,0	7,5	12,5	**43,5**
Salt-water +ice	15*0,8*10=120	70	56	2,1	3,2	5,4	**22,5**

M_c for chilling (from 10 to 0°C) = (M_s* Cp_s * ΔT) / H_f = (15*0,762*(20-0))/80,
M_c for storage = (1,5%*M_s*10 days * 80kcal/kg) * ratio of ice in the cooling agent,
M_c = mass of cooling agent (kg), M_s = mass of shrimp (15 kg),
Cp_s = specific heat used for shrimp (80 kcal/kg), ΔT = 10°C, ΔH_f = latent heat of fusion

On days 0, 1, 4 and 6 of storage, corresponding to days 3, 4, 7 and 9 after catch, duplicate samples were taken from each lot of the four different groups of shrimp stored in the different conditions. The samples were submitted to microbiological, chemical, physical and sensory analysis.

Table 2: Experimental groups and sampling plan.

Group	Type of ice	Ratio of shrimp to ice	Draining during storage	Storage temp.	Sampling days 0	1	4	6
ICE/+	Flake ice	1:1.5	Yes	1.5±0.4°C	Day0	ICE/+1	ICE/+4	ICE/+6
LIQ/+	Liquid ice	1:2.9	Yes	1.5±0.4°C	Day0	LIQ/+1	LIQ/+4	LIQ/+6
S-ICE/-	Salt-water (30%) + ice (70%)	1:1.5	No	-1.5±0.3°C	Day0	S-ICE/-1	S-ICE/-4	S-ICE/-6
LIQ/-	Liquid ice	1:2.9	No	-1.5±0.3°C	Day0	LIQ/-1	LIQ/-4	LIQ/-6

3.2 Sensory evaluation

A Quality Grading Scheme was used to evaluate the quality of whole shrimp (Table 3). Duplicate samples from each of the four storage conditions were taken at regular intervals (on days 0, 1, 4 and 6 of storage) for each group and placed in two clean transparent glass containers, after 20 min the assessment was carried out under room temperature and adequate fluorescent light. The samples were coded with a random three digit number. The panalists were not aware of the number of storage days of the shrimp and did not know which two containers were the same group prior to assessment. The panel constituted of eight members who had been trained in evaluating quality of shrimp and the characteristic sensory attributes.

Table 3: Score sheet for quality grading scheme of whole shrimp (IFL 2003).

Score / Grading	Description
5 Excellent	Colour is dark red to bright pink. Roes are blue-green (copper). Strong seaweedy, marine odour. Strong sweet shrimp taste.
4 Good	Colour is natural light pink. Roes are blue-green (copper). Weak characteristic shrimp odour. Weak sweet shrimp taste.
3 Moderate	Marine/shrimp odour is diminishing, weak "fishy odour", even slight ammonia. Colour is natural light pink with grey-greenish or yellowish discoloration. Roes are light green. Taste is natural not sweet to weak "fishy taste".
2 Borderline----Clearly not Fresh	Weak ammonia odour. Colour is natural light pink with grey-greenish or yellowish discoloration. Roes are discoloured. Blackening on the head can be spotted. Distinct fishy taste with bitter aftertaste.
1 Unfit Spoiled	Ammonia odour. Colour is natural light pink with grey-greenish or yellowish discoloration. Roes are Dark. The blackening on the head is extensive. Spoiled, taste with strong, bitter aftertaste.

3.3 Protein measurement

Protein content in shrimp meat was determined by the Kjeldahl method (ISO 1997). A sample of 5.00 g was digested in sulphuric acid in presence of copper as a catalyst. Thereafter, the sample was placed in distillation unit, 2400 Kjeltec Auto Sample System. The acid solution was made alkaline by a sodium hydroxide solution. The ammonia was distilled into boric acid and the acid was simultaneously titrated with diluted H_2SO_4. The nitrogen content was multiplied by the factor 6.25 to get the ratio of crude protein.

3.4 Salt measurement

Salt content in the shrimp meat was determined using the potentiometric method (AOAC 1995). Soluble chloride was extracted from the sample with water containing nitric acid. The chloride content of the solution was titrated with silver nitrate and the end point was determined potentiometrically.

3.5 Fat measurement

Fat content in shrimp meat was determined by the method of AOCS Official Method Ba-3-38 (1997). The sample was extracted with petroleum ether, boiling range 40-60°C. The extraction apparatus was 2025 Soxtec Avanti Automatic System.

3.6 Water measurement

Water content in shrimp meat was determined according to the method ISO 6496 (1999). The sample was heated in a heating oven at 103°C +/- 2°C for four hours. Water corresponds to the weight loss.

3.7 Water-holding capacity (WHC) measurement

Water-holding capacity (WHC) of peeled whole shrimp was measured by modified centrifuge method reported by Eide *et al*. (1982). Water removed during centrifuge was drained through the nylon membrane in the sample holder, and collected in the bottom of the centrifuge tube (50ml). The conditions were: around 3.5 g sample (the individual numbers of peeled whole shrimp was 2 or 3); centrifuge time, 5 min at 3500 rpm; at 10 °C. The sample holder was weighed before and after centrifuge for determination of weight loss of the sample. The water-holding capacity was expressed as following:
Water-holding capacity (%) = ((weight of the sample × water content % of the sample - weight loss of the sample) / (weight of the sample × water content % of the sample)) × 100.

3.8 TVB-N and TMA measurement

Total volatile basic nitrogen (TVB-N) and trimethylamine (TMA) were determined using steam distillation in the minced shrimp tissue, followed by titration method (AOAC 1990). The TVB-N was performed through direct distillation into boric acid using a Kjeldahl-type distillatory (Struer TVN) (Malle and Poumeyrol, 1989), the acid was titrated with diluted H_2SO_4 solution. To determine TMA the same method was used as for TVB-N but adding 20 ml of 35% formaldehyde to the distillation flask to block the primary and secondary aminess, an alkaline binding mono-and di-amine, TMA being the only volatile and measurable amine (Malle and Poumeyrol 1989). The TVB-N and TMA content was expressed in mgN/100g shrimp tissue.

3.9 pH measurement

pH was measured using a calomel electrode (SE 104) pH meter (Knick-Portamess 913 (X) pH meter, Germany, Berlin). Glass calomel electrode was dipped into minced shrimp meat at room temperature.

3.10 Texture measurement

A compression test was carried out. The sample was placed on the baseplate and compressed two times by a platen attached to the drive system using a texture analyzer (TA-XT2I Texture Analyzer, Stable Micro Systems) as seen in Figure 2. The texture analyzer equipped with a 75 mm diameter rounded head probe and a 5 kN load cell was used; the cross speed was set at 0.80 mm/s, the post test speed was 10.00 mm/s, and a 100 g constant force. In order to ensure no cracking of sample, the compression was limited to 50% of the sample height on the basis of preliminary trials. The trigger force was set at 5 g and the registration rate to 200 PPS (registrations s). Five measurements in five individuals from each lot were carried out.

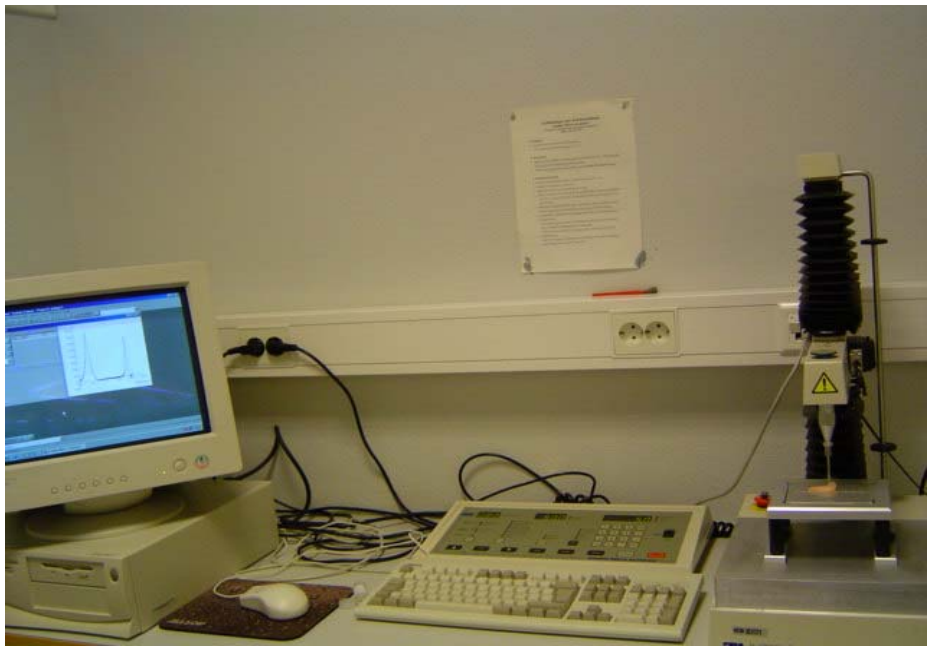

Figure 2: Texture analyzer (TA-XT2I Texture Analyzer) used to measure texture change in shrimp.

3.11 Electronic Nose measurement

Electronic nose measurements were performed using an electronic nose called FreshSense (Figure 3), developed by the Icelandic Fisheries Laboratories (IFL) and Bodvaki (Maritech, Iceland) (Olafsdottir *et al*. 2002). The instrument consists of a glass container closed with a plastic lid, an aluminum sensor box fastened to the lid, and a personal computer running a measurement program. The sensor box contains four

different electrochemical gas sensors (Dräger, Germany, CO, H_2S, and SO_2; City Technology, U.K., NH_3) and a temperature sensor. Electronic, and A/D converter, and a microprocessor to read the measurements and send them to the computer are also in the box. A fan is positioned in the container to ensure gas circulation. The measurement technique was reported earlier by Olafsdottir *et al*. (1997a). 500 g. of shrimp were analyzed; the measurement time was 5 min and temperature was 7-9^{o}C during the measurements.

Figure 3: Electronic nose FreshSense used to measure quality change of shrimp.

3.12 Bacteriological test

The total viable counts (TVC) was performed according to the Compendium of Methods for the Microbiological Examination of foods published by the American Public Health Association (APHA 1992). The samples of whole shrimp for bacteriological analysis to estimate total viable counts (TVC) were first minced. This procedure was then followed by weighing 25 g of each the minced sample, homogenizing it in 225 g of dilution buffer. 1 ml of the primary 1/10 suspension was then withdrawn and decimal dilutions were prepared in dilution buffer. Total viable counts were done on agar containing 0.5% NaCl by pour plate and incubated at 22°C for 72 hrs for psycrotrophic bacteria. The conventional "pour-plate" method was used. Plates showing colony numbers of 25 to 250 were then selected for counting. The number of colonies counted thus constituted the total viable counts (TVC).

3.13 Data analysis

The data, including instrumental texture parameters, sensory score and water-holding capacity value, was tested using analysis of variance (ANOVA) to analyze if a difference existed within a group and among groups during the storage time, and to show the Duncan's Multiple-Comparison Test. Linear equation and the correlation coefficients (R) of some indicators such as total volatile bases nitrogen (TVB-N), trimethylamine (TMA), total viable counts (TVC), water content, salt content and electronic nose measurement parameters were calculated. Principal component analysis (PCA), which was conducted in the statistical program Unscrambler (Version 7.5, CAMO ASA, Oslo, Norway), was performed to study the main tendencies of the variation among the measurement variable and to evaluate if the various analytical techniques applied were comparable to evaluate quality. In all cases, significance levels were set at 95% ($P<0.05$).

4 RESULTS

4.1 Basic characteristics of the sample and temperature change during storage

Upon its arrival at the laboratory, the size and proximate composition of the shrimp were measured. The mean weight and length of the shrimp were 5.1±0.6g and 9.2±0.7cm, respectively. The moisture 81.1%; crude protein 17.4%; crude fat 0.4%; salt (NaCl) 0.7%.

The average temperature of the cold storage room, in which the two liquid iced groups were stored, was -1.5±0.3°C. Another cold storage room, in which the two iced groups were stored, was of 1.5±0.4°C. The centre temperature in each bin holding sample during storage is shown in Figure 4.

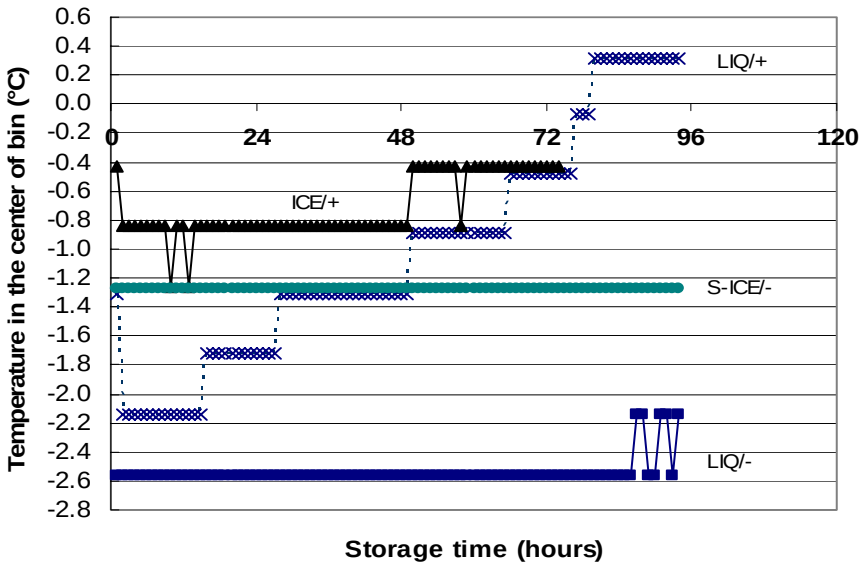

Figure 4: The centre temperature in each bin holding sample during storage. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

Compared to traditional ice storage, the liquid ice could maintain lower temperature and chill shrimp more rapidly. A gradual increase of centre temperature was found in the group LIQ/+, which may be explained by the decrease of liquid ice with time because the melted ice was drained continuously. The increase in temperature was also noticed with storage time for the traditional ice storage ICE/+. The melted ice was also drained for that sample and the influence of the higher storage temperature of the cooling room is obvious for these two groups LIQ/+ and ICE/+.

4.2 Sensory evaluation

The average sensory score calculated for each sample formed a linear relationship with storage time for each group/lot (Figure 5). The shrimp stored in liquid ice at -1.5°C scored significantly higher ($P<0.05$) than other lots throughout the 6-day storage period.

The lowest score was awarded to the shrimp group (ICE/+) stored in ice at 1.5°C throughout the whole storage period. The appearance of the four sample groups of shrimp on the 6th day of storage, are shown in Figure 6.

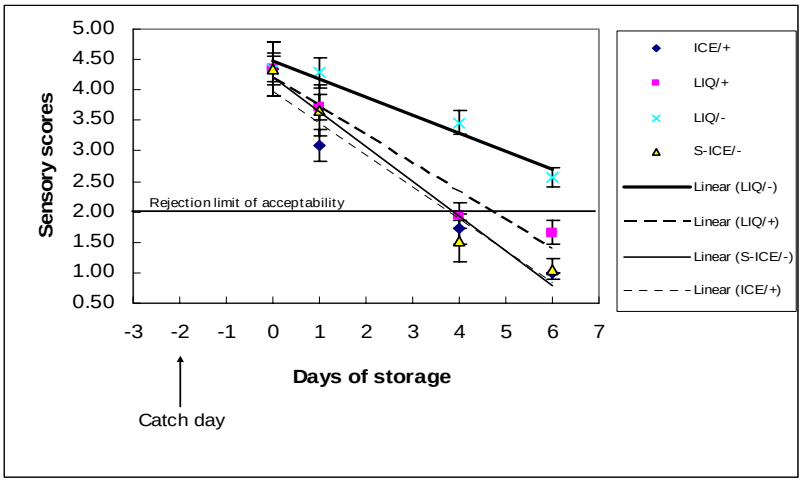

Figure 5: Sensory scores of shrimp stored in different cooling conditions. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

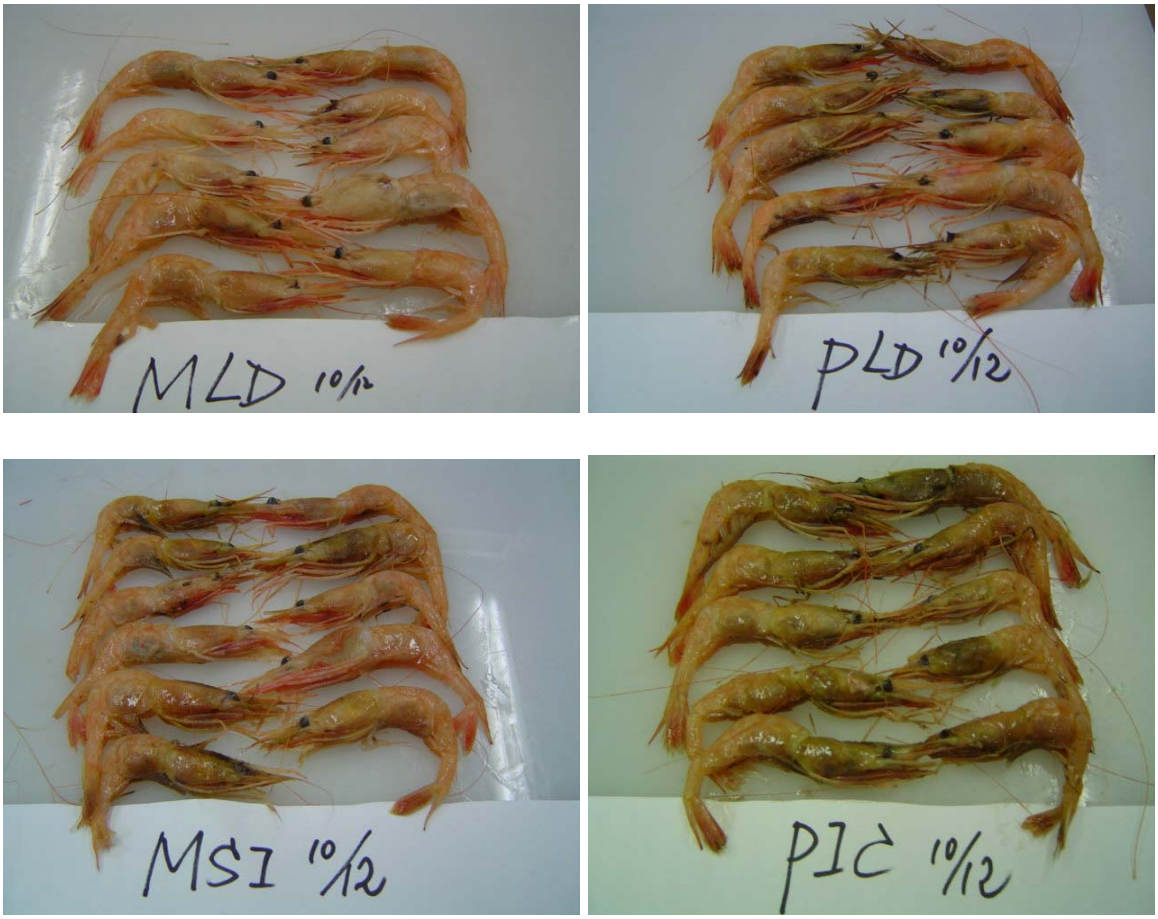

Figure 6: Appearance of shrimp stored in different cooling conditions on the day 6th of storage. PIC (ICE/+): flake ice at 1.5°C; PLD (LIQ/+): liquid ice at 1.5°C; MSI (S-ICE/-): salt-water + ice -1.5°C; MLD (LIQ/-): liquid ice at -1.5°C.

These pictures show the differences in appearance of shrimp among groups. The larger the black discoloration on the surface of shrimp, the lower the quality of the shrimp. As seen on the figure the sample labelled PIC (ICE/+) appears to have the highest proportion of discoloration. This is in agreement with the sensory analysis showing this sample had the lowest Grading Scheme scores for freshness evaluation throughout the storage.

4.3 TVB-N and TMA

Total volatile basic nitrogen (TVB-N) value of 33.5 mg/100g whole shrimp was measured at the beginning of storage and on day 1,4 and 6 (Figure 7). The effect of different storage type and conditions on TMA formation in shrimp is shown in Figure 8.

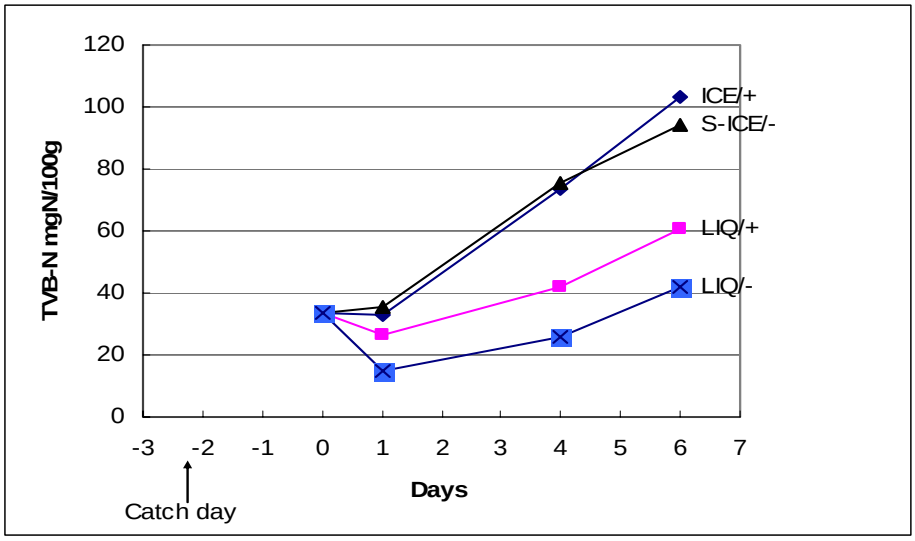

Figure 7: Total volatile basic nitrogen (TVB-N) (mgN/100g) formation of shrimp stored in different cooling conditions during 6 days storage period. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

On day 1 the TVB-N values for LIQ/- and LIQ/+ had lowered from day 0. The values for ICE/+ and S-ICE/- changed very little during day 1. Then the TVB-N value started to increase but TVB-N for LIQ/- was always the lowest. However in the other two groups, ICE/+ and S-ICE/- that showed the highest TVB-N value, the TVB-N value increased to more than 70 mg/100g the fourth day of storage.

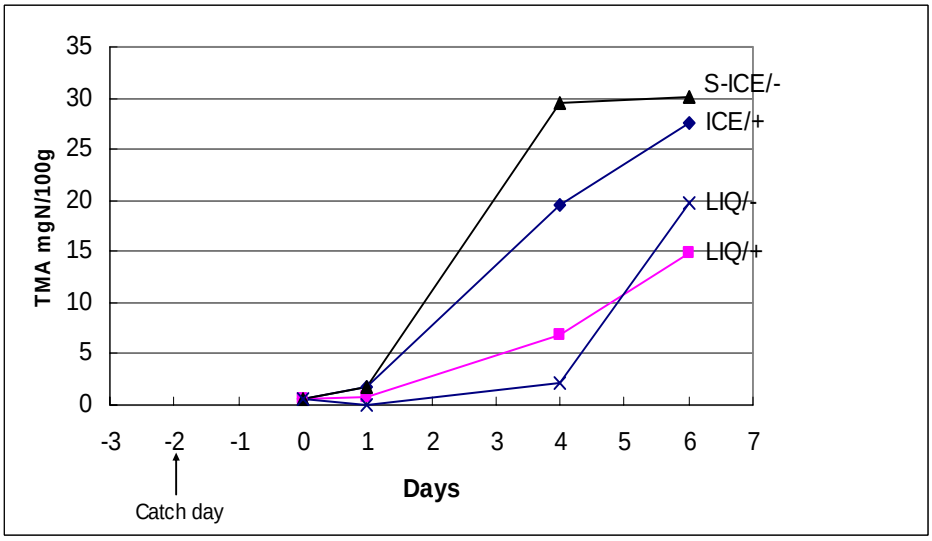

Figure 8: Trimethylamine (TMA) (mgN/100g) formation of shrimp stored in different cooling conditions during 6 days storage period. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

Initial TMA value of the sample was 0.5 mgN/100g on day 0 when the shrimp arrived at laboratory (Figure 8). TMA formation gradually increased over the storage period with the exception of liquid ice group (LIQ/-) at lower temperature (-1.5°C) where TMA was reduced to 0 mgN/100g on day 1 and then a short lag period before TMA began to increase steadily in the following storage days.

4.4 pH measurement

Mean pH measurements over the period of iced or liquid iced storage are shown in Figure 9. The initial pH of the shrimp was 7.41 upon its arrival. Results show that the increases of pH value were rapid in the two samples that had been stored in ice at 1.5°C and in salt-water ice at -1.5°C, and reached 8.26 and 8.20, respectively (Figure 9). However, the changes were small in samples stored in liquid ice at -1.5°C. In the end of storage the pH was 7.98.

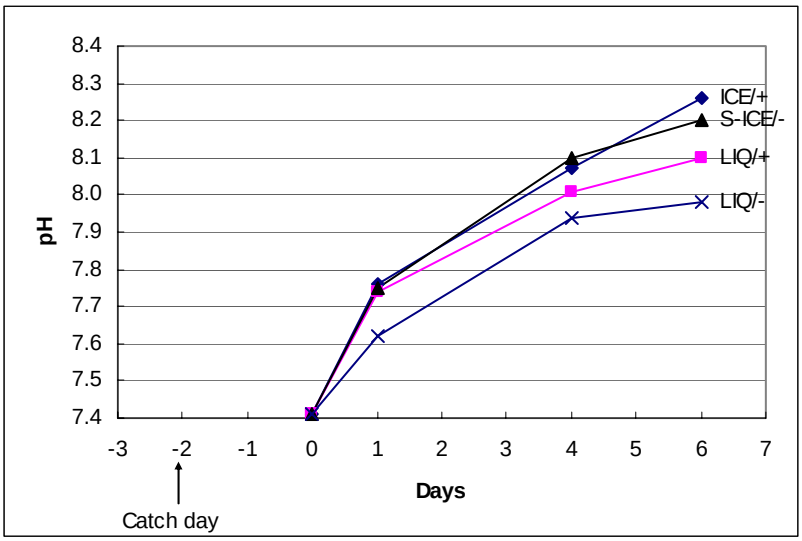

Figure 9: Changes of pH value of the shrimp stored in different conditions. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

4.5 Water content

The effects of storage type and storage time on the changes in absolute water content during the storage period of whole shrimp are shown in Figure 10.

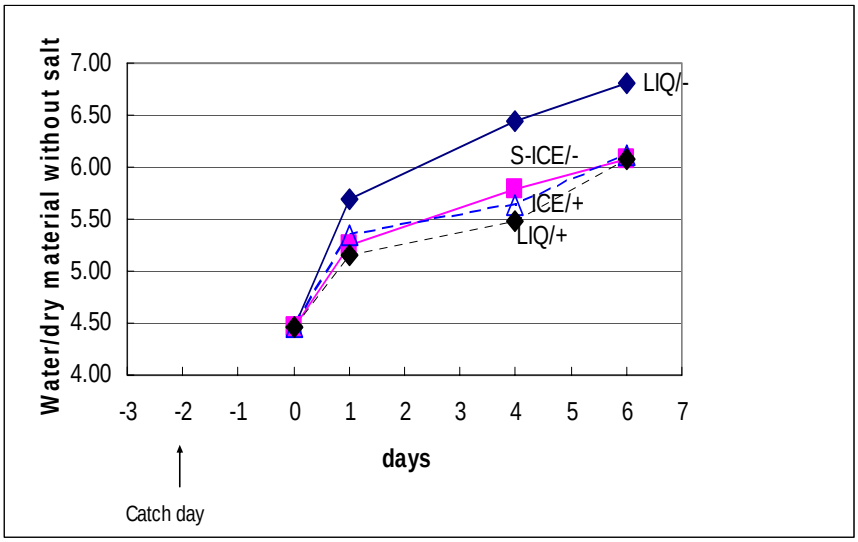

Figure 10: Changes of water content of shrimp stored in different conditions during the storage period. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

Although no significant increases were found in relative moisture content between each group, the results show that the water content increased gradually with storage time from initial 81.1% gradually to around 85% in all the groups during the storage period (data not shown). The findings were similar to the report that presented an increase in the weight of headed cod in fluid ice by 3% to 6% over a 10-h period (Huidobro *et al*. 2002.

4.6 Salt content

The results from the salt (NaCl) content analysis of shrimp muscle under various storage types are shown in Figure 11.

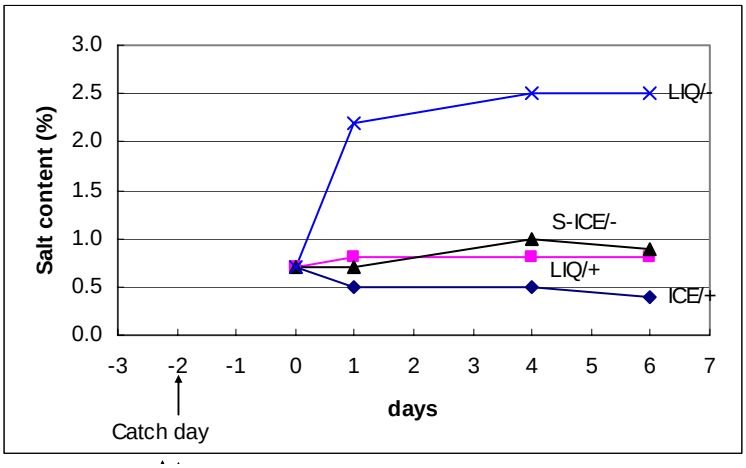

Figure 11: Changes of salt content of shrimp stored in different conditions during the storage period. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

The salt content increased slowly in shrimp that were stored in LIQ/+ or S-ICE/-. A rapid increase in salt content for the sample group stored in liquid ice at -1.5°C was found. However, the salt content in iced shrimp decreased slowly during the storage period.

4.7 Water-holding capacity (WHC)

The water-holding capacity of the shrimp is shown in Figure 12. It is evident that the water-holding capacity of the shrimp after storage is lower than that for the raw shrimp before storage.

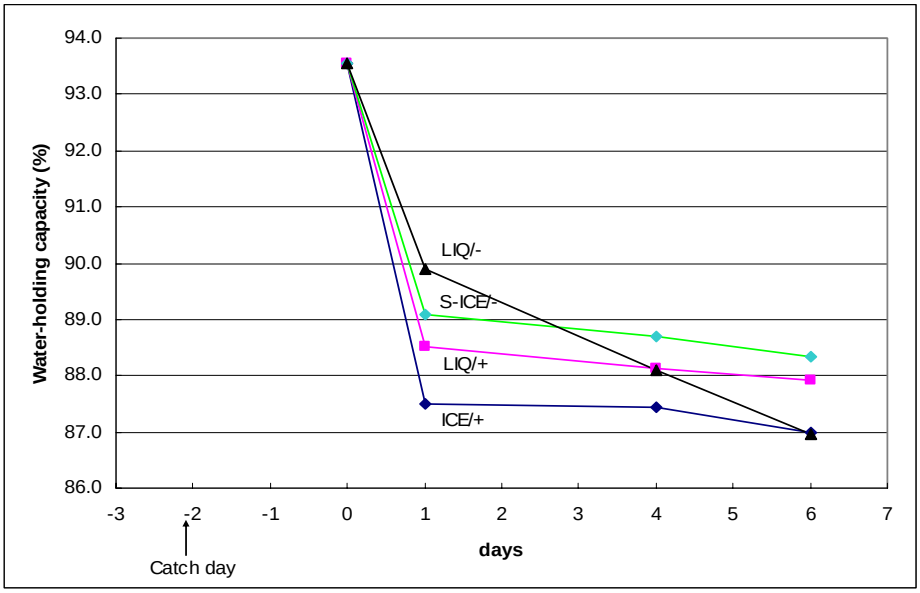

Figure 12: Changes of water-holding capacity of shrimp stored in different conditions during the storage period. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

The water-holding capacity for each lot decreased with the storage time. No significant differences in water-holding capacity were found between the four groups and between each lot within two groups that were the S-ICE/- and LIQ/+. The ICE/+ group had a significant difference ($P<0.05$) in water-holding capacity between the raw material (Day0) and other lots (ICE/+1, ICE/+4, ICE/+6). The LIQ/- group also indicated a significance difference between Day0, LIQ/-1 and LIQ/-6, between Day0, LIQ/-1 and LIQ/-4 (Figure 12 and Appendix). The results of correlation analysis show a good correlation between water-holding capacity and water content ($r=0.87$, shown in Table 4). This means that the higher the water content, the stronger water-holding capacity in shrimp. However, excessive water content, for instance, water content exceeded 2% in sample stored in liquid ice at -1.5°C (Figure 10), resulted in decrease in water-holding capacity in shrimp (Figure 12).

4.8 Texture measurement

The hardness, springiness, cohesiveness and resilience measured by Texture Analyzer are shown in Figure 13-16, respectively. The results of texture measurement and from variance analysis (ANOVA) show that the variation of texture parameters, including hardness, springiness, resilience and cohesiveness, was small for the various storage groups or storage times.

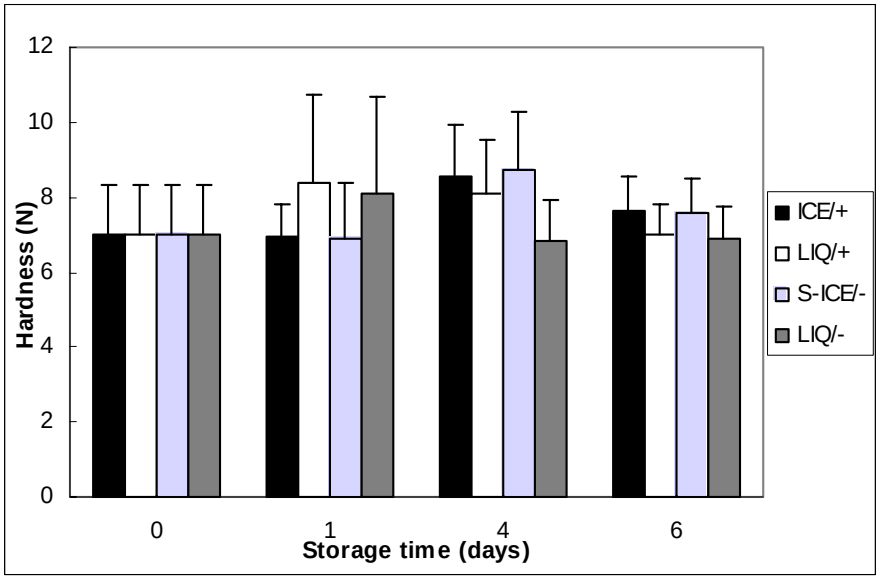

Figure 13: Hardness (N) of shrimp stored in different conditions. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

No evident differences of hardness were found during storage between the groups and lots in each group (Appendix). It seems that hardness is not influenced by the storage type and time.

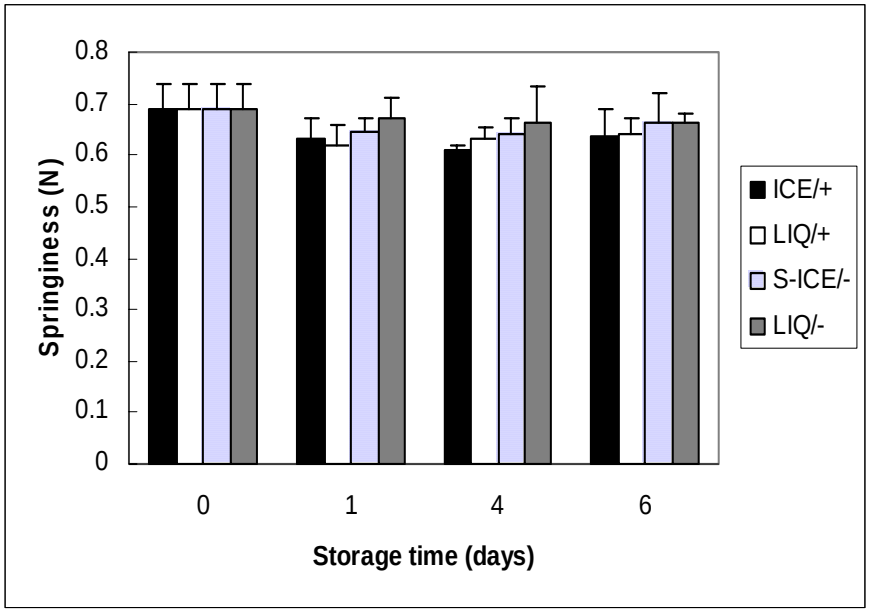

Figure 14: Springiness (%) of shrimp stored in different conditions. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

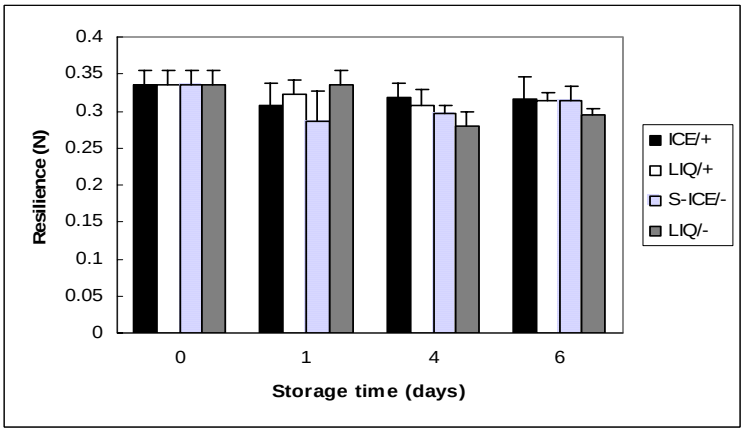

Figure 15: Resilience of shrimp stored in different conditions. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

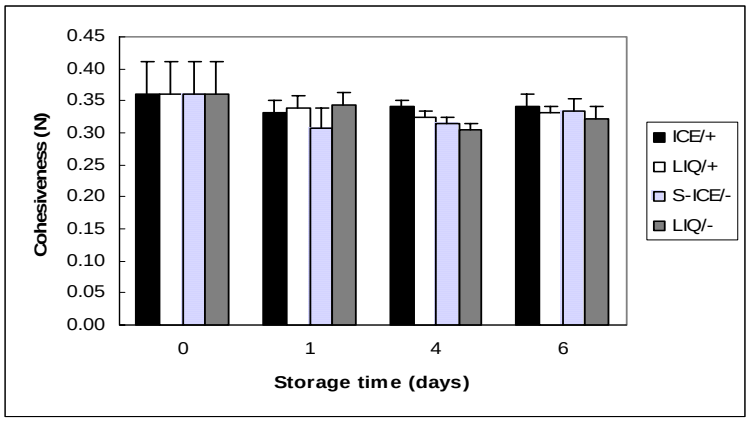

Figure 16: Cohesiveness of shrimp stored in different conditions. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

Although some incidental individual significant differences existed in some groups or lots, there were no obvious regular trends in the changes in the springiness, resilience and cohesiveness of the shrimp stored under different conditions. In general, springiness and cohesiveness decreased at the beginning of storage and increased again later, although the extent and step of changes were different in the four groups. Therefore, it is necessary to develop better methods. Similar results were shown by Huidobro *et al.* (2001) who reported no differences between compression tests applied on gilthead seabream killed by immersion in liquid ice and by immersion in ice plus water.

4.9 Electronic nose measurement

The responses of CO and NH_3 sensors were highest and most sensitive among the sensors of the electronic nose, for the samples stored at different conditions (Figure 17 and 18). The responses of the H_2S and SO_2 sensors were not accounted for in the report due to their low responses towards all the sample groups during storage. This indicates that the development of sulfur compounds is of little importance during storage of shrimp under these conditions.

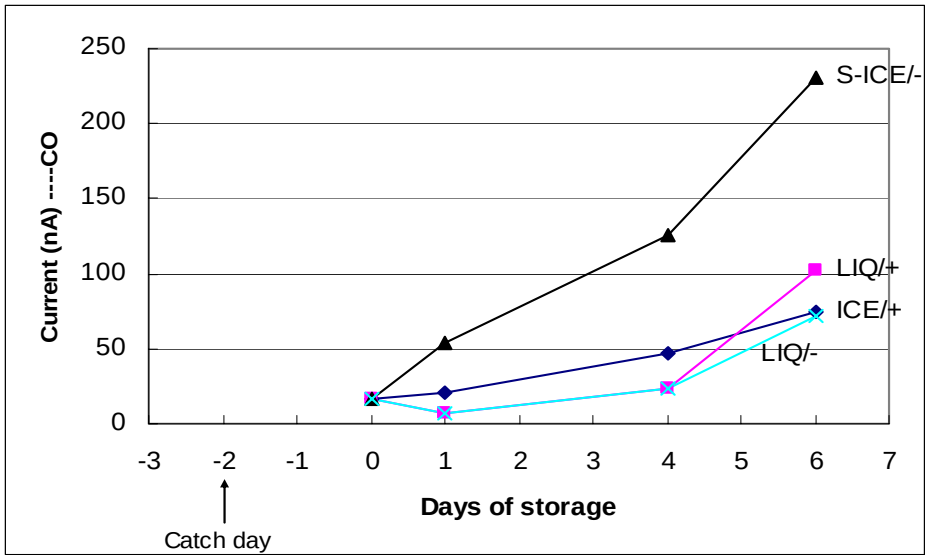

Figure 17: Responses of CO sensors to the shrimp stored in different condition. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

The CO formation in S-ICE/- was very rapid but the other treatments (LIQ/-, LIQ/+, ICE/+) showed less CO formation. The highest CO value was measured at about 230 nA.

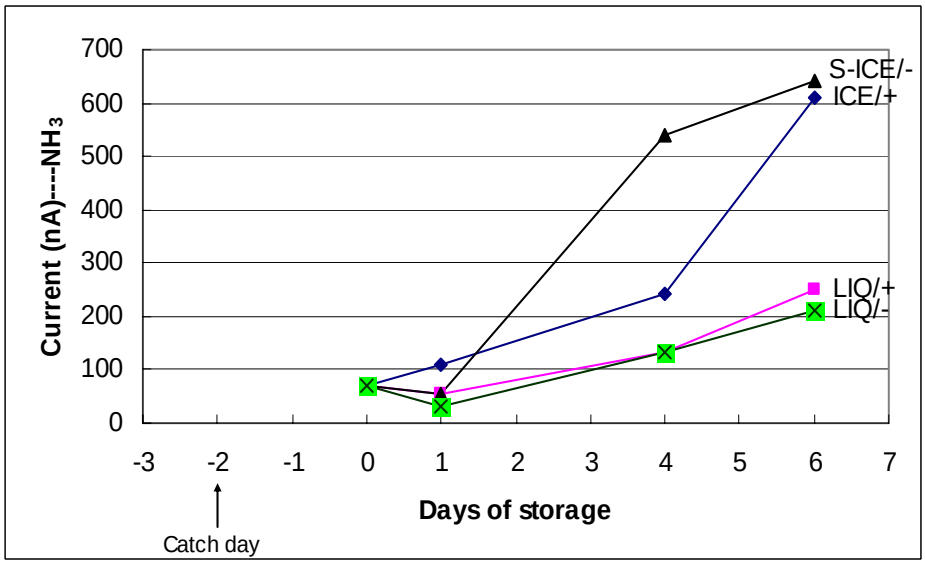

Figure 18: Responses of NH_3 sensors to the shrimp stored in different condition. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

The NH_3 formation was different from CO formation where S-ICE/- and ICE/+ showed much higher values than LIQ/- and LIQ/+. The NH_3 value on day 1 decreased for ICE/+, LIQ/- and LIQ/+ but started to increased after day 1.

4.10 Bacteriological test

Bacteriological changes as monitored during storage are shown in Figure 19. TVC in shrimp in S-ICE/- and ICE/+ lots increased steadily. The microbiological growth rate in shrimp chilled in ice is faster than in the other three groups during the storage period. From the initial level of 2.4×10^5 cfu/g (TVC) increased to 3×10^8 cfu/g in the sample stored in ice at 1.5°C by the end of the storage period when TVC was 10^6 cfu/g in the sample stored in liquid ice at -1.5°C and the TVC levels in other lots were 1.7×10^7 cfu/g and 6.4×10^7 cfu/g, respectively.

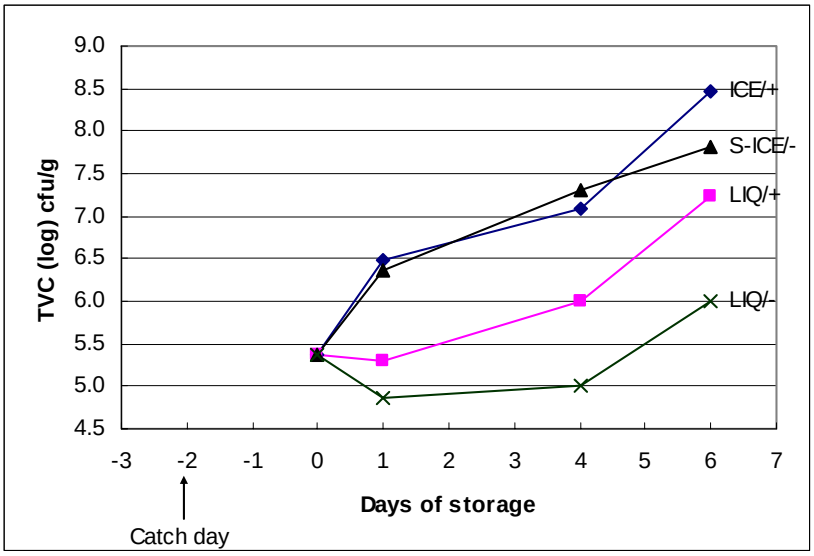

Figure 19: Changes in total viable counts (TVC) in shrimp during storage. ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C.

4.11 Correlation between indicators

Table 4 shows the correlation coefficients between the parameters measured i.e. salt content, water content, pH, TMA, TVB-N, TVC, electronic nose (CO and NH_3 responses) , sensory score, texture (H, S, C, R), water-holding capacity (WHC) and W/D. The yellow colour highlights where good correlations are found. The texture parameters do not show any correlations to the other quality indicators measured.

Table 4: Correlation (r) between parameters for quality assessment of shrimp.

	Salt	Water	pH	TMA	TVB-N	TPC	CO	NH3	Sensory score	H	S	C	R	WHC	W/D
Salt	1.00														
Water	0.08	1.00													
pH	-0.12	0.93	1.00												
TMA	-0.16	0.72	0.84	1.00											
TVB-N	-0.46	0.65	0.81	0.92	1.00										
TPC	-0.57	0.67	0.78	0.84	0.95	1.00									
CO	-0.14	0.56	0.65	0.81	0.75	0.71	1.00								
NH3	-0.25	0.66	0.80	0.94	0.94	0.86	0.83	1.00							
Sensory score	0.35	-0.82	-0.94	-0.89	-0.90	-0.88	-0.71	-0.85	1.00						
H	-0.25	0.03	0.22	0.31	0.27	0.15	0.05	0.25	-0.28	1.00					
S	0.51	-0.36	-0.42	-0.16	-0.25	-0.35	0.09	-0.07	0.41	-0.50	1.00				
C	-0.36	-0.41	-0.34	-0.07	0.08	0.01	-0.14	-0.03	0.13	0.17	0.17	1.00			
R	-0.32	-0.40	-0.33	-0.09	0.03	-0.04	-0.14	-0.06	0.13	0.35	0.11	0.95	1.00		
WHC	-0.05	-0.87	-0.76	-0.44	-0.37	-0.45	-0.23	-0.35	0.61	-0.08	0.63	0.48	0.47	1	
W/D	0.58	0.85	0.69	0.53	0.31	0.27	0.40	0.43	-0.49	-0.16	0.02	-0.49	-0.49	-0.71	1

H: Hardness; S: Springiness; C: Cohesiveness; R: Resilience; W/D: water/dry material; WHC: water-hold capacity

4.12 PCA (principal component analysis) analysis

The data from the various measurements used to monitor quality in shrimp stored under different conditions was analyzed by principal component analysis (PCA) as shown in Figures 20-21.

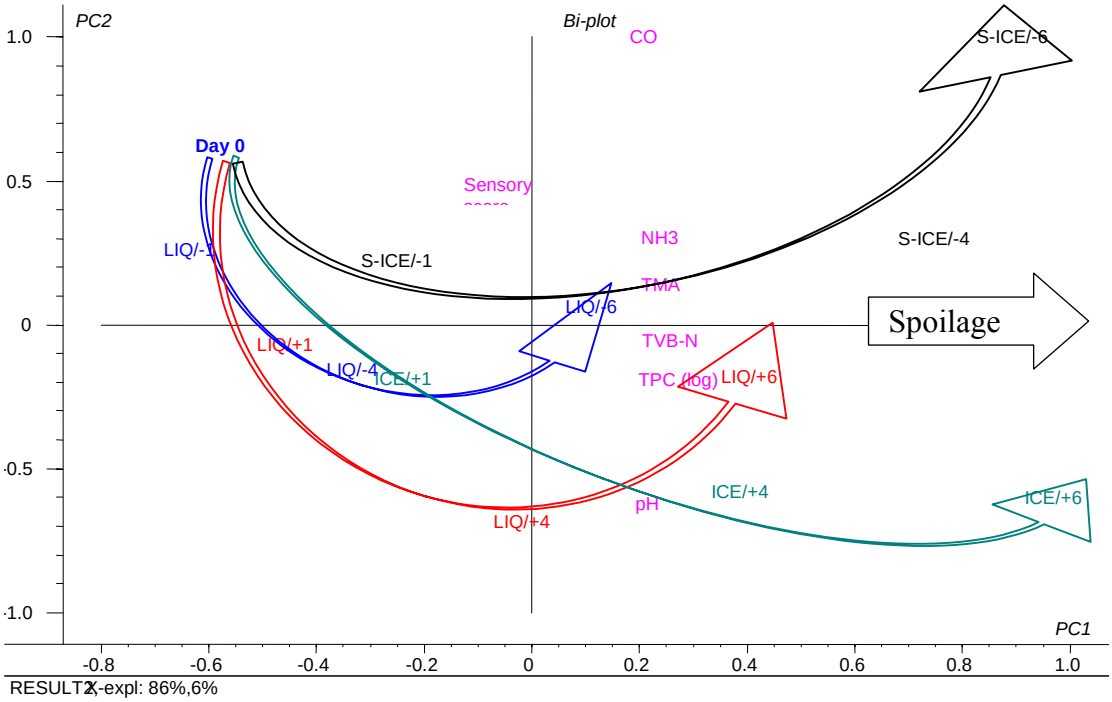

Figure 20: Bi-plot for PCA of measured main data. Sample scores are labeled with the storage condition and days of storage (ICE/+: flack ice at 1.5°C; LIQ/+: liquid ice at 1.5°C; S-ICE/-: salt-water + ice -1.5°C; LIQ/-: liquid ice at -1.5°C). Loadings of variables include TVB-N, TMA, TVC, pH, sensory score and FreshSenSe measurements (CO and NH_3).

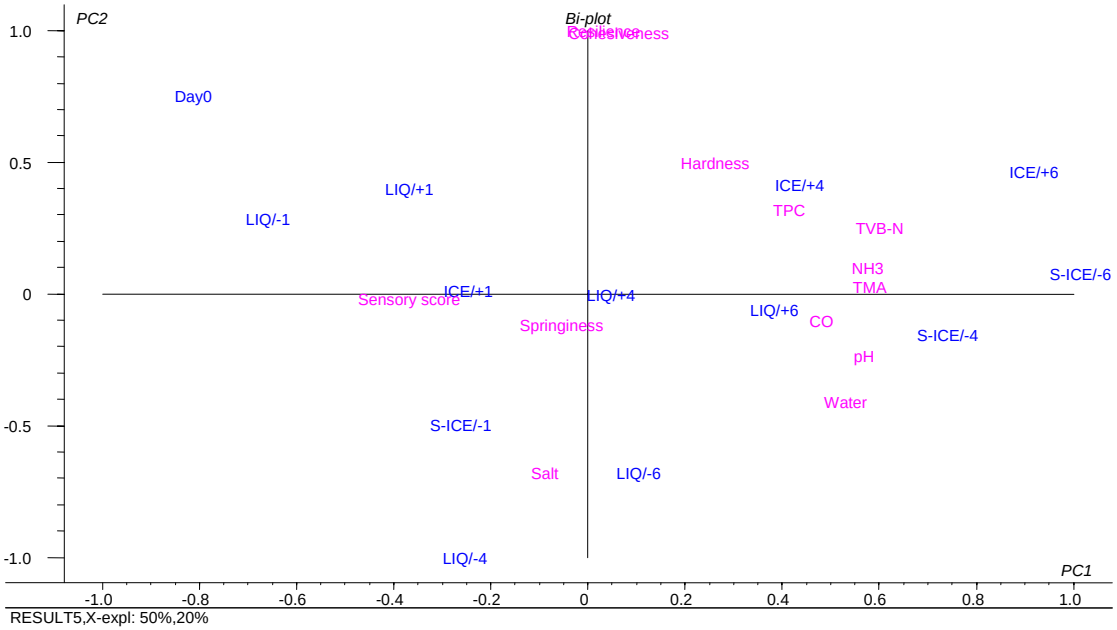

Figure 21: Bi-plot for PCA of all the measured data. Sample scores are labelled with the storage condition and days of storage (ICE/+: flack ice at 1.5^{o}C; LIQ/+: liquid ice at 1.5^{o}C; S-ICE/-: salt-water + ice -1.5^{o}C; LIQ/-: liquid ice at -1.5^{o}C). Loadings of variables include TVB-N, TMA, TVC, pH, water, salt, texture (hardness, springiness, resilience, cohesiveness), sensory score and FreshSense measurements (CO and NH_3).

Figure 20 shows the PCA scores and loadings plots of all samples and data from TVB-N, TMA, TVC, pH, sensory scores and CO, NH_3 response of electronic nose FreshSense measurement. The *X*-axis is the first principal component (PC1) that explains 86% of the variance in the data set and PC2 explains 6%, a total of 92% of the variation in the data set is explained by the model. The x axis is explaining the spoilage level of the samples and when the salt and water content and the texture parameters are added less variation in the data set is explained by the model as shown in Figure 21.

Figure 21 shows the PCA for the same samples and variables as Figure 20 but also includes the water and salt content and the texture parameters. The first principal component (PC1) explains 50% of the variation in the data set and PC2 explains 20%. This shows that the texture parameters and the salt and water content do not provide additional information to the model to explain the variation in the spoilage level of the samples.

5 DISCUSSIONS

5.1 Sensory evaluation

Sensory analysis of whole shrimp revealed that significant differences ($P<0.05$) were found between the groups or lots (Appendix). The results from the analysis of variance (ANOVA) showed that every lot (LIQ/-1, LIQ/-4 and LIQ/-6) in the group LIQ/- (in which shrimp stored in liquid ice at -1.5^{o}C) presented a significant difference with other lots in other groups. No significant difference was found between LIQ/-1 and Day0 or between LIQ/+1 and Day0 which indicates that very little changes occurring in the shrimp stored in liquid ice after 1 day of storage. Contrasting results were found in other groups (Appendix). Moreover, increasing differences were evident as shown in Figure 20 between the LIQ/- group and other groups with the storage time. The shrimp stored in liquid ice at -1.5^{o}C had an overall higher score that means higher quality or lower spoilage than other groups throughout a 6-day storage period, although some assessors reported a little lower colour score as a result of a slight whiteness in the liquid ice group. The lowest score was awarded to the shrimp stored in ice at 1.5^{o}C throughout the storage (ICE/+). The results of sensory evaluation also showed that the sensory scores decreased linearly with storage time and the linear equations and correlation coefficients are the following respectively:

ICE/+ group, $y=-0.53x+3.98$, $R^2=0.95$; LIQ/+ group, $y=-0.47x+4.20$, $R^2=0.95$;
S-ICE/- group, $y=-0.57x+4.21$, $R^2=0.97$; LIQ/- group, $y=-0.30x+4.48$, $R^2=0.96$.

These equations show that the shrimp stored in LIQ/- had the slowest spoilage rate as seen by the lowest slopes value (0.2973) in the linear equation for sensory scores vs. days. However, the shrimp stored in S-ICE/- appeared to have the fastest spoilage rate (slope value is 0.5699). This is similar to the result shown by TMA, CO and NH_3 responses of electronic nose measurement.

5.2 TVB-N and TMA change

Total volatile basic nitrogen (TVB-N) value of 33.5 mg/100g whole shrimp was found at the beginning of storage (Figure 7). The high initial value of TVB-N is most likely because not enough ice was present to maintain constant temperature during the delayed transport and the temperature of the raw material had reached 4 °C when it arrived at the laboratory. The high temperature encourages the growth of spoilage bacteria (initial count of 2.4×10^5 cfu/g). The microbial degradation of TMAO and deamination of amino acids resulting in the formation of TMA and ammonia, respectively, is evidenced by high initial values of TVB-N. Putrescine- and cadaverine-forming bacteria in shrimp can also grow at 0^{o}C and contribute to amine formation (Lakshmanan *et al*. 2002). After 1 day storage lower value was observed for the TVB-N and a delay in the onset of TVB-N production in the groups stored in liquid ice (LIQ/- or LIQ/+). The group LIQ/-, showed lower TVB-N levels on day 1 of storage and a longer lag phase before resuming increases than the other groups. The increase of TVB-N in shrimp stored in liquid ice was slower than the other two lots stored in ice or salt-water ice. The results suggest that the growth

of the main spoilage-causing microorganism was restrained by the liquid ice. Similar results were shown by the bacteria measurements (Figure 19).

A comparison of the rates of TMA formation during 6 days of storage revealed that salt-water iced group (S-ICE/-) and iced group (ICE/+), in which TMA values exceeded 10 mgN/100g, spoiled earlier than other two groups where TMA level remained below 10 mgN/100g until day four of storage. The extent of increase in TVB-N and TMA of shrimp stored in liquid ice at -1.5^{o}C were considerably smaller than for sample groups stored under other conditions (S-ICE/-, ICE/+).

5.3 Change in pH of whole shrimp

The pH of shrimp meat gives some valuable information about its quality change. Significant differences were noticed from Figure 9 and the results of statistical analysis. There was a continued increase in pH for all sample groups, probably due metabolism of microorganisms producing alkaline compounds like amines formed by deamination of amino acids (Huss, 1988, Jackson *et al*. 1997). The initial post mortem pH varies with species, catching ground and season. Usually pH decreases during anaerobic formation of lactic acids during the first hours after death, but microbial metabolism leads to an increase in pH during storage time. This is in good agreement with Krishnakumar *et al*. (1985) who showed reduction of total nitrogen in fish stored in ice sea-water and ice because some compounds contained nitrogen were leached out. The pH changes, showed good correlation with sensory and microbiological results. The pH changes also reflected TVB-N and TMA accumulation and indicated the spoilage progress.

5.4 CO and NH_3 responses of electronic nose measurement

Results from electronic nose measurements indicate that response of NH_3 sensor can be used to evaluate the shrimp quality in a similar way as TVB-N, TMA and TVC (Table 4). The rapid onset of NH_3 production at low bacterial cell densities indicates that autolysis may be causing the production during the first day of storage and the rapid increase in the rate of production during following days of storage indicates a bacterial contribution as reported by Lakshmanan *et al*. (2002), who found that the amine-forming bacterial population in fresh shrimp was slightly higher (10^2 cfu.g^{-1}) than in fish. Olafsdottir *et al*. (1997a, 2002) reported that the NH_3 response of electronic nose measurement gave the best prediction of TVB in capelin raw material and was similar to the information provided by TMA for redfish. The CO sensor showed lower responses than the NH_3 sensor, but similar overall trends for all the storage groups except for the group stored in ICE/+ on day 6. It can be speculated that the decline in the CO sensor could be explained by specific spoilage flora utilizing different substrates for their growth and thus form different volatile degradation compounds. It is well known that the development of microbial metabolites changes because of competition of the microflora for available substrates. However, this can not be confirmed because only one measurement was done. The lower responses of the CO sensor indicating lower spoilage level of samples of shrimp stored in ice is not in accordance with results from other indicators that were TVC, TVB-N and TMA in the trial. This should be studied further in combination with

microbial studies to identify the specific spoilage bacteria in shrimp under these conditions.

5.5 Total viable count (TVC)

Figure 19 shows the log TVC observed during the storage period for the four groups of shrimp. Initial TVC of the shrimp was 2.4×10^5 cfu/g (Figure 19). A decrease in bacterial total numbers to 7.2×10^4 cfu/g and 2.0×10^5 cfu/g was observed in the two groups stored in liquid ice at -1.5°C and at 1.5°C respectively after one day of storage. The initial reduction in the total bacteria can be explained because of cold shock (Ingram 1951). The growth was first resumed after a lag phase of at least 24 h, and the slowest bacterial growth was found in the sample stored in liquid ice at -1.5°C compared to other groups. The results are in good agreement with the report presented by Lakshmanan *et al.* (2002). After 6 days of storage, total viable counts in the shrimp stored in ice at 1.5°C (ICE/+) were two logarithmic units higher than the group LIQ/- stored in liquid ice at -1.5°C. The TVC exceeded spoilage level of 10^7 cfu/g (Capell *et al*. 1997) with the exception of the group stored in liquid ice at -1.5°C at the end of the trial. Actually, the extension of shelf-life of the shrimp stored in liquid ice at -1.5°C was attributed to delayed microbial growth.

Other reports have shown that liquid ice can flow freely and surround the entire sample resulting in rapid cooling and less damage of the samples. Bacterial growth was hindered and high quality was maintained (Optimar 2003). Similar studies performed by the Canadian Centre for Fisheries Innovation (CCFI) show that liquid ice (Optim-Ice) performed better than regular icing methods when icing Snow Crab (Optimar 2003).

Shewan (1961) has shown that TVC was lower in fish stored in ice sea-water than in ice stored fish, and explained his results by faster initial cooling and lower storage temperature during the ice sea-water storage. During storage microbiological growth rate in shrimp in LIQ/- group is the lowest in all these groups. It may be affected by rapid cooling and lower storage temperature (below 0°C) and better covering in shrimp in liquid ice. Similar results were seen in the Optimar (2003) information.

5.6 Correlation analysis

The correlation coefficients between sensory score and other parameters are minus value because the sensory scores have decreasing values with storage while the values for the other quality parameters are increasing with storage. The fact that there are very good correlations between these parameters such as pH, TMA, TVB-N, TVC, NH_3 and sensory score illustrates that pH, NH_3 response of electronic nose measurement and sensory evaluation, which are rapid and practical methods, can be used as quality indicators of northern shrimp. Among pH, NH_3 response of electronic nose measurement and sensory evaluation, NH_3 response of electronic nose measurement is the best feasible quality indicator for the shrimp because of its simple and rapid operation. Followed by pH because of its simple and rapid measurement although slight lower veracity. The last one is sensory evaluation which has advantage of assessing quality with no facilities. However, the assessors who had been trained in evaluating quality of shrimp and the characteristic sensory attributes are needed.

5.7 PCA analysis

Principal component analysis (PCA) for all of the samples, which was conducted in the statistical program Unscrambler (Version 7.5, CAMO ASA, Oslo, Norway), was performed to study the main tendencies of the variation among the measurement variable and to evaluate if the various analytical techniques applied were comparable to evaluate quality. PCA was also done to study the main trend in the data and to illustrate the effect of the different storage types on the quality and spoilage level of shrimp. Most of the latent variables methods used in multivariate data analysis are in one way or another based on PCA (Wold *et al*. 1987). The PCA method provides a simple and efficient way for graphically describing systematic variation in complex data structures. Principal component analysis (PCA) is a tool for identifying relationships in complex analytical data by comparing data in more than one dimension. The main objective is to detect structure in the relationship between measured parameters and experimental factors. It has been used to transform a number of possibly correlated variables into a (smaller) number of uncorrelated variables called principal components. The first component explains as much of the variability in the data as possible, then the second component will account for as much of the remaining variability as possible, etc.

It can be seen that the first PC1 represents the quality spoilage level of the sample with the increasing storage time from left to right along PC1 (Figure 20, 21). Group ICE/+6 and S-ICE/-6, even ICE/+4 and S-ICE/-4 are located to the right in the diagram, while LIQ/-6 is just located to the middle. The result indicates that the shrimp stored in LIQ/- tend to spoil later than the other groups, the shrimp stored in ICE/+ spoil first, and the shrimp stored in S-ICE/- spoil sooner than the others. The sample LIQ/-6 had high loadings for the salt which indicate high level of salt content in the sample. The sample ICE/+4 and ICE/+6 had high loadings for the TVC and pH value. The texture parameters measured in shrimp contribute very little to PC1 and do not appear to change with storage time (Figure 21). The NH_3 response, TMA, TVB-N and TVC are located close to each other on the plot (Figure 20, 21), illustrating that these indicators keep high correlation and give the similar information that can indicate the quality of the shrimp, the findings

are in agreement with the results from analysis of variance (r=0.84 ~ 0.94, Table 4). Olafsdottir *et al.* (2002) reported that the CO response was highly correlated to the sensory score (QIM) for redfish under all storage conditions, and that the response of the NH_3 sensor and TMA measurement give similar information and have very good correlation for redfish stored in ice. It is interesting that both the CO and NH_3 sensors show higher responses towards the S-ICE/- group compared with the ICE/+ group which is in agreement with the result of TMA analysis showing higher values for the S-ICE/- group.

This should be studied further in combination with microbial studies to identify the specific spoilage bacteria in shrimp under these conditions. These results suggest that metabolites from TMA producing bacteria contribute to the responses of the CO and NH_3 sensors. These could be Pseudomonas species that are known to also produce volatile ketones, aldehyde and esters that the CO sensor can detect (Huss 1995). The PCA plot (Figure 20) shows that the loading of the CO sensor appears to contribute to the positioning of the S-ICE/- group on the upper half of the plot indicating a different spoilage pattern for that group, perhaps because of conditions that favour the growth of a different specific spoilage bacteria compared with the other groups.

6 CONCLUSIONS

Comparison of sensory, chemical, microbiological and physical quality parameters of shrimp, stored in ice at 1.5^{o}C (ICE/+), in liquid-ice at 1.5^{o}C (LIQ/+), in liquid-ice at 1.5^{o}C (LIQ/-), and in salt-water ice at -1.5^{o}C (S-ICE/-), showed that S-ICE/- did not extend the shelf-life of shrimp as compared to ICE/+, whereas LIQ/+ and LIQ/- with the rapid cooling and lower temperature and better covering delayed the rate of quality deterioration and extended the shelf-life, especially LIQ/- gave the longest shelf-life and the best quality shrimp.

Application of liquid ice storage decreased the rate of TVB-N and TMA formation and delayed the growth of microorganism compared to salt-water iced or iced storage. Rate of production of both TVB-N or TMA and total viable counts (TVC) in shrimp stored in ice or in salt-water ice was always higher than other two groups, which were stored in liquid ice. The shelf-life of shrimp stored in liquid ice at -1.5^{o}C was extended compared to others storage conditions according to the indicators which were TVB-N, TMA, pH, TVC, NH_3 response of electronic nose measurement and sensory evaluation. Shrimp stored in liquid ice at -1.5^{o}C showed higher sensory score and indicated higher quality than other iced types throughout the storage period, although a slight loss of the characteristic colour was observed.
Good correlation existed between TVB-N and TMA (r=0.92), TVB-N and NH_3 (r=0.94), NH_3 and TMA (r=0.94) and TVB-N and sensory evaluation (r=0.90). Good correleation was also found between TVC and the following parameters TVB-N (r=0.95), TMA (r=0.84), NH3 (r=0.86), sensory evaluation (r=0.88) and between pH and sensory evaluation (r=0.94).

NH_3 response of electronic nose measurement correlates well with traditional quality evaluation technique (TVB-N, TMA and TVC) and the CO sensor may give further information about the presence of specific spoilage bacteria. This indicates that electronic nose measurements can be used effectively to monitor quality and onset of spoilage of shrimp.

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APPENDIX

Analysis of Variance Report (Term significant at 95%, α=0.05)

1. WHC: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Group	3	20,81317	6,937723	1,50	0,265172	0,298529
S(A)	12	55,58823	4,632352			
Total (Adjusted)	15	76,40139				
Total	16					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	16	90,10438		22,52609
A: Group				
LIQ/-1	4	91,5875	1,076145	69,06141
S-ICE/-1	4	90,5075	1,076145	67,98141
ICE/+1	4	88,4325	1,076145	65,9064
LIQ/+1	4	89,89	1,076145	67,36391

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
ICE/+1	4	88,4325	
LIQ/+1	4	89,89	
S-ICE/-1	4	90,5075	
LIQ/-1	4	91,5875	

2. Sensory score: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Group	3	5,65625	1,885417	12,90	0,000018*	0,999388
S(A)	28	4,09375	0,1462054			
Total (Adjusted)	31	9,75				
Total	32					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	32	3,6875	0,4609375	
A: Group				
LIQ/-1	8	4,28125	0,1351875	3,820313
S-ICE/-1	8	3,65625	0,1351875	3,195313
ICE/+1	8	3,09375	0,1351875	2,632813
LIQ/+1	8	3,71875	0,1351875	3,257813

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
ICE/+1	8	3,09375	S-ICE/-1, LIQ/+1, LIQ/-1
S-ICE/-1	8	3,65625	ICE/+1, LIQ/-1
LIQ/+1	8	3,71875	ICE/+1, LIQ/-1
LIQ/-1	8	4,28125	ICE/+1, S-ICE/-1, LIQ/+1

3. WHC: Analysis of Variance Table

Source	Sum of	Mean	Prob	Power

Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxx	3	5,293719	1,764573	0,21	0,885434	0,079315
S(A)	12	99,38853	8,282377			
Total (Adjusted)	15	104,6822				
Total	16					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	16	89,03812		22,25953
A: Groupxx				
LIQ/-4	4	89,0325	1,438956	66,77297
S-ICE/-4	4	89,7775	1,438956	67,51797
ICE/+4	4	88,1675	1,438956	65,90797
LIQ/+4	4	89,175	1,438956	66,91547

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
ICE/+4	4	88,1675	
LIQ/-4	4	89,0325	
LIQ/+4	4	89,175	
S-ICE/-4	4	89,7775	

4. Sensory score: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxx	3	18,81836	6,272787	35,97	0,000000*	1,000000
S(A)	28	4,882813	0,1743862			
Total (Adjusted)	31	23,70117				
Total	32					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	32	2,164063	0,2705078	
A: Groupxx				
LIQ/-4	8	3,46875	0,1476424	3,198242
S-ICE/-4	8	1,53125	0,1476424	1,260742
ICE/+4	8	1,71875	0,1476424	1,448242
LIQ/+4	8	1,9375	0,1476424	1,666992

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
S-ICE/-4	8	1,53125	LIQ/-4
ICE/+4	8	1,71875	LIQ/-4
LIQ/+4	8	1,9375	LIQ/-4
LIQ/-4	8	3,46875	S-ICE/-4, ICE/+4, LIQ/+4

5. Hardness: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxx	3	11,29872	3,766239	1,98	0,155272	0,419554
S(A)	17	32,3389	1,902288			
Total (Adjusted)	20	43,63762				
Total	21					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	21	8,057905		1,534598
A: Groupxx				
LIQ/-4	5	6,8256	0,6168125	5,291002
S-ICE/-4	5	8,7538	0,6168125	7,219202

ICE/+4	5	8,564	0,6168125	7,029402
LIQ/+4	6	8,083167	0,5630702	6,548568

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/-4	5	6,8256	
LIQ/+4	6	8,083167	
ICE/+4	5	8,564	
S-ICE/-4	5	8,7538	

6. Springiness: Analysis of Variance Table

Source Term	DF	Sum of Squares (Alpha=0,05)	Mean Square	F-Ratio	Prob Level	Power
A: Groupxx	3	7,101219E-03	2,367073E-03	3,31	0,045426*	0,647164
S(A)	17	1,217173E-02	7,159843E-04			
Total (Adjusted)	20	1,927295E-02				
Total	21					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	21	0,6359524	0,1211651	
A: Groupxx				
LIQ/-4	5	0,6618	1,196649E-02	0,5406349
S-ICE/-4	5	0,6408	1,196649E-02	0,5196349
ICE/+4	5	0,6092	1,196649E-02	0,4880349
LIQ/+4	6	0,6326666	1,092386E-02	0,5115016

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
ICE/+4	5	0,6092	LIQ/-4
LIQ/+4	6	0,6326666	
S-ICE/-4	5	0,6408	
LIQ/-4	5	0,6618	ICE/+4

7. Cohesiveness: Analysis of Variance Table

Source Term	DF	Sum of Squares (Alpha=0,05)	Mean Square	F-Ratio	Prob Level	Power
A: Groupxx	3	3,317738E-03	1,105913E-03	8,00	0,001529*	0,967328
S(A)	17	0,0023495	1,382059E-04			
Total (Adjusted)	20	5,667238E-03				
Total	21					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	21	0,3208095	6,108095E-02	
A: Groupxx				
LIQ/-4	5	0,3054	5,257488E-03	0,2443191
S-ICE/-4	5	0,3138	5,257488E-03	0,252719
ICE/+4	5	0,34	5,257488E-03	0,278919
LIQ/+4	6	0,3235	4,799407E-03	0,262419

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/-4	5	0,3054	ICE/+4
S-ICE/-4	5	0,3138	ICE/+4

LIQ/+4	6	0,3235	ICE/+4
ICE/+4	5	0,34	LIQ/-4, S-ICE/-4, LIQ/+4

8. Resilience: Analysis of Variance Table

Source Term	DF	Sum of Squares (Alpha=0,05)	Mean Square	F-Ratio	Prob Level	Power
A: Groupxx	3	4,226805E-03	1,408935E-03	5,13	0,010413*	0,846469
S(A)	17	4,668433E-03	2,746137E-04			
Total (Adjusted)	20	8,895238E-03				
Total	21					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	21	0,3008095	5,722699E-02	
A: Groupxx				
LIQ/-4	5	0,279	7,410988E-03	0,221773
S-ICE/-4	5	0,2968	7,410988E-03	0,239573
ICE/+4	5	0,3178	7,410988E-03	0,260573
LIQ/+4	6	0,3081667	6,765276E-03	0,2509397

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/-4	5	0,279	LIQ/+4, ICE/+4
S-ICE/-4	5	0,2968	
LIQ/+4	6	0,3081667	LIQ/-4
ICE/+4	5	0,3178	LIQ/-4

9. WHC: Analysis of Variance Table

Source Term	DF	Sum of Squares (Alpha=0,05)	Mean Square	F-Ratio	Prob Level	Power
A: Groupx	3	9,205869	3,068623	0,59	0,634139	0,137715
S(A)	12	62,57483	5,214569			
Total (Adjusted)	15	71,78069				
Total	16					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	16	88,13937		22,03484
A: Groupx				
LIQ/-6	4	87,46	1,141772	65,42516
S-ICE/-6	4	89,1425	1,141772	67,10766
ICE/+6	4	87,3525	1,141772	65,31766
LIQ/+6	4	88,6025	1,141772	66,56766

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
ICE/+6	4	87,3525	
LIQ/-6	4	87,46	
LIQ/+6	4	88,6025	
S-ICE/-6	4	89,1425	

10. Sensory score: Analysis of Variance Table

Source Term	DF	Sum of Squares (Alpha=0,05)	Mean Square	F-Ratio	Prob Level	Power
A: Groupx	3	12,59961	4,19987	19,83	0,000000*	0,999998
S(A)	28	5,929688	0,2117746			

Total (Adjusted)	31	18,5293			
Total	32				

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	32	1,570313		
	0,1962891			
A: Groupx				
LIQ/-6	8	2,5625	0,1627016	2,366211
S-ICE/-6	8	1,0625	0,1627016	
	0,8662109			
ICE/+6	8	1	0,1627016	
	0,8037109			
LIQ/+6	8	1,65625	0,1627016	1,459961

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
ICE/+6	8	1	LIQ/+6, LIQ/-6
S-ICE/-6	8	1,0625	LIQ/+6, LIQ/-6
LIQ/+6	8	1,65625	ICE/+6, S-ICE/-6, LIQ/-6
LIQ/-6	8	2,5625	ICE/+6, S-ICE/-6, LIQ/+6

11. Hardness: Analysis of Variance Table

Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupx	3	2,752417	0,9174722	1,18	0,342413	0,266855
S(A)	19	14,72837	0,7751772			
Total (Adjusted)	22	17,48079				
Total	23					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	23	7,297131		1,265981
A: Groupx				
LIQ/-6	5	6,8714	0,3937454	5,605419
S-ICE/-6	6	7,594167	0,3594387	6,328186
ICE/+6	6	7,656	0,3594387	6,390019
LIQ/+6	6	6,996	0,3594387	5,730019

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/-6	5	6,8714	
LIQ/+6	6	6,996	
S-ICE/-6	6	7,594167	
ICE/+6	6	7,656	

12. Springiness: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupx	3	2,749613E-03	9,165377E-04	0,48	0,702833	0,128182
S(A)	19	0,0366003	1,926332E-03			
Total (Adjusted)	22	3,934991E-02				
Total	23					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	23	0,6507826		
	0,1132623			
A: Groupx				
LIQ/-6	5	0,6622	0,0196282	
	0,5489377			

S-ICE/-6		6	0,6618333	1,791801E-02	0,548571
ICE/+6		6	0,6376666	1,791801E-02	
		0,5244043			
LIQ/+6		6	0,6433333	1,791801E-02	0,530071

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
ICE/+6	6	0,6376666	
LIQ/+6	6	0,6433333	
S-ICE/-6	6	0,6618333	
LIQ/-6	5	0,6622	

13. Cohesiveness: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power
	(Alpha=0,05)					
A: Groupx	3	1,192286E-03	3,974285E-04	1,45	0,259706	0,321569
S(A)	19	5,207367E-03	2,740719E-04			
Total (Adjusted)	22	6,399652E-03				
Total	23					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	23	0,3324348		
	5,772899E-02			
A: Groupx				
LIQ/-6	5	0,3206	7,403674E-03	0,262871
S-ICE/-6	6	0,334	6,758599E-03	0,276271
ICE/+6	6	0,3413333	6,758599E-03	
	0,2836044			
LIQ/+6	6	0,3318333	6,758599E-03	
	0,2741044			

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/-6	5	0,3206	
LIQ/+6	6	0,3318333	
S-ICE/-6	6	0,334	
ICE/+6	6	0,3413333	

14. Resilience: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power
	(Alpha=0,05)					
A: Groupx	3	1,705619E-03	5,685396E-04	1,13	0,360434	0,256766
S(A)	19	9,524033E-03	5,012649E-04			
Total (Adjusted)	22	1,122965E-02				
Total	23					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	23	0,3105652		
	0,0538942			
A: Groupx				
LIQ/-6	5	0,2944	1,001264E-02	
	0,2405058			
S-ICE/-6	6	0,3145	9,140249E-03	
	0,2606058			
ICE/+6	6	0,317	9,140249E-03	
	0,2631058			
LIQ/+6	6	0,3136667	9,140249E-03	
	0,2597724			

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/-6	5	0,2944	
LIQ/+6	6	0,3136667	
S-ICE/-6	6	0,3145	
ICE/+6	6	0,317	

15. WHC: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxx	3	95,053	31,68433	4,89	0,019068*	0,785394
S(A)	12	77,7713	6,480942			
Total (Adjusted)	15	172,8243				
Total	16					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	16	89,3725		22,34312
A: Groupxxx				
Day0	4	93,5375	1,272885	71,19437
ICE/+1	4	88,4325	1,272885	66,08938
ICE/+4	4	88,1675	1,272885	65,82437
ICE/+6	4	87,3525	1,272885	65,00938

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
ICE/+6	4	87,3525	Day0
ICE/+4	4	88,1675	Day0
ICE/+1	4	88,4325	Day0
Day0	4	93,5375	ICE/+6, ICE/+4, ICE/+1

16. Sensory-score: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxx	3	52,84961	17,61654	215,49	0,000000*	1,000000
S(A)	28	2,289063	8,175223E-02			
Total (Adjusted)	31	55,13867				
Total	32					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	32	2,539063		
	0,3173828			
A: Groupxxx				
Day0	8	4,34375	0,1010892	4,026367
ICE/+1	8	3,09375	0,1010892	2,776367
ICE/+4	8	1,71875	0,1010892	1,401367
ICE/+6	8	1	0,1010892	
	0,6826172			

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
ICE/+6	8	1	ICE/+4, ICE/+1, Day0
ICE/+4	8	1,71875	ICE/+6, ICE/+1, Day0
ICE/+1	8	3,09375	ICE/+6, ICE/+4, Day0
Day0	8	4,34375	ICE/+6, ICE/+4, ICE/+1

17. Hardness: Analysis of Variance Table

Source	Sum of	Mean	Prob	Power

Term	DF	Squares	Square	F-Ratio	Level	
	(Alpha=0,05)					
A: Groupxxx	3	9,176114	3,058705	2,27	0,111335	0,489230
S(A)	20	26,92393	1,346196			
Total (Adjusted)	23	36,10004				
Total	24					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	24	7,485833		1,257639
A: Groupxxx				
Day0	8	7,029125	0,4102128	5,771486
ICE/+1	5	6,9342	0,5188827	5,676561
ICE/+4	5	8,564	0,5188827	7,306362
ICE/+6	6	7,656	0,4736729	6,398362

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
ICE/+1	5	6,9342	
Day0	8	7,029125	
ICE/+6	6	7,656	
ICE/+4	5	8,564	

18. Springiness: Analysis of Variance Table

Source		Sum of	Mean		Prob	Power
Term	DF	Squares	Square	F-Ratio	Level	
	(Alpha=0,05)					
A: Groupxxx	3	2,292863E-02	7,642875E-03	4,61	0,013137*	0,817213
S(A)	20	3,317721E-02	1,65886E-03			
Total (Adjusted)	23	5,610583E-02				
Total	24					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	24	0,6480833		
	0,1070684			
A: Groupxxx				
Day0	8	0,689375	1,439992E-02	
	0,5823066			
ICE/+1	5	0,6334	1,821461E-02	
	0,5263316			
ICE/+4	5	0,6092	1,821461E-02	
	0,5021316			
ICE/+6	6	0,6376666	1,662759E-02	
	0,5305983			

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
ICE/+4	5	0,6092	Day0
ICE/+1	5	0,6334	
ICE/+6	6	0,6376666	Day0
Day0	8	0,689375	ICE/+4, ICE/+6

19. Cohesiveness: Analysis of Variance Table

Source		Sum of	Mean		Prob	Power
Term	DF	Squares	Square	F-Ratio	Level	
	(Alpha=0,05)					
A: Groupxxx	3	3,149667E-03	1,049889E-03	3,89	0,024302*	0,741699
S(A)	20	5,396833E-03	2,698417E-04			
Total (Adjusted)	23	0,0085465				
Total	24					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect

All	24	0,34575		
	0,0572743			
A: Groupxxx				
Day0	8	0,36125	5,807771E-03	
	0,3039757			
ICE/+1	5	0,332	7,346314E-03	
	0,2747257			
ICE/+4	5	0,34	7,346314E-03	
	0,2827257			
ICE/+6	6	0,3413333	6,706237E-03	0,284059

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
ICE/+1	5	0,332	Day0
ICE/+4	5	0,34	
ICE/+6	6	0,3413333	
Day0	8	0,36125	ICE/+1

20. Resilience: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxx	3	2,639333E-03	8,797778E-04	1,48	0,249922	0,330939
S(A)	20	0,01188	0,000594			
Total (Adjusted)	23	1,451933E-02				
Total	24					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	24	0,3211667		
	5,321667E-02			
A: Groupxxx				
Day0	8	0,335	8,616844E-03	
	0,2817833			
ICE/+1	5	0,3074	1,089954E-02	
	0,2541833			
ICE/+4	5	0,3178	1,089954E-02	
	0,2645833			
ICE/+6	6	0,317	9,949874E-03	
	0,2637833			

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
ICE/+1	5	0,3074	
ICE/+6	6	0,317	
ICE/+4	5	0,3178	
Day0	8	0,335	

21. WHC: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxxx	3	59,18652	19,72884	3,27	0,058965	0,596759
S(A)	12	72,36945	6,030787			
Total (Adjusted)	15	131,556				
Total	16					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	16	90,30125		22,57531
A: Groupxxxx				
Day0	4	93,5375	1,227883	70,96219
LIQ/+1	4	89,89	1,227883	67,31469

LIQ/+4		4	89,175	1,227883	66,59969
LIQ/+6		4	88,6025	1,227883	66,02719

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/+6	4	88,6025	
LIQ/+4	4	89,175	
LIQ/+1	4	89,89	
Day0	4	93,5375	

22. Sensory score: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power
	(Alpha=0,05)					
A: Groupxxxx	3	41,81836	13,93945	62,37	0,000000*	1,000000
S(A)	28	6,257813	0,2234933			
Total (Adjusted)	31	48,07617				
Total	32					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	32	2,914063	0,3642578	
A: Groupxxxx				
Day0	8	4,34375	0,1671426	3,979492
LIQ/+1	8	3,71875	0,1671426	3,354492
LIQ/+4	8	1,9375	0,1671426	1,573242
LIQ/+6	8	1,65625	0,1671426	1,291992

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/+6	8	1,65625	LIQ/+1, Day0
LIQ/+4	8	1,9375	LIQ/+1, Day0
LIQ/+1	8	3,71875	LIQ/+6, LIQ/+4, Day0
Day0	8	4,34375	LIQ/+6, LIQ/+4, LIQ/+1

23. Hardness: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power
	(Alpha=0,05)					
A: Groupxxxx	3	9,41386	3,137954	1,38	0,275680	0,313014
S(A)	21	47,67176	2,270084			
Total (Adjusted)	24	57,08562				
Total	25					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	25	7,55028		1,220724
A: Groupxxxx				
Day0	8	7,029125	0,5326918	5,808401
LIQ/+1	5	8,4098	0,6738077	7,189076
LIQ/+4	6	8,083167	0,6150994	6,862443
LIQ/+6	6	6,996	0,6150994	5,775276

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/+6	6	6,996	
Day0	8	7,029125	
LIQ/+4	6	8,083167	
LIQ/+1	5	8,4098	

24. Springiness: Analysis of Variance Table

Source Term	DF	Sum of Squares (Alpha=0,05)	Mean Square	F-Ratio	Prob Level	Power
A: Groupxxxx	3	1,939442E-02	6,464806E-03	4,66	0,011938*	0,826195
S(A)	21	2,911174E-02	1,386273E-03			
Total (Adjusted)	24	4,850616E-02				
Total	25					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	25	0,65056		0,103359
A: Groupxxxx				
Day0	8	0,689375	1,316374E-02	0,586016
LIQ/+1	5	0,6186	1,665097E-02	0,515241
LIQ/+4	6	0,6326666	1,520018E-02	0,5293077
LIQ/+6	6	0,6433333	1,520018E-02	0,5399743

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/+1	5	0,6186	Day0
LIQ/+4	6	0,6326666	Day0
LIQ/+6	6	0,6433333	Day0
Day0	8	0,689375	LIQ/+1, LIQ/+4, LIQ/+6

25. Cohesiveness: Analysis of Variance Table

Source Term	DF	Sum of Squares (Alpha=0,05)	Mean Square	F-Ratio	Prob Level	Power
A: Groupxxxx	3	5,655607E-03	1,885202E-03	9,83	0,000298*	0,991853
S(A)	21	4,028633E-03	1,918397E-04			
Total (Adjusted)	24	9,68424E-03				
Total	25					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	25	0,34052	5,419133E-02	
A: Groupxxxx				
Day0	8	0,36125	4,896934E-03	0,3070587
LIQ/+1	5	0,3382	6,194186E-03	0,2840087
LIQ/+4	6	0,3235	5,654492E-03	0,2693087
LIQ/+6	6	0,3318333	5,654492E-03	0,277642

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/+4	6	0,3235	Day0
LIQ/+6	6	0,3318333	Day0
LIQ/+1	5	0,3382	Day0
Day0	8	0,36125	LIQ/+4, LIQ/+6, LIQ/+1

26. Resilience: Analysis of Variance Table

Source Term	DF	Sum of Squares (Alpha=0,05)	Mean Square	F-Ratio	Prob Level	Power
A: Groupxxxx	3	2,883193E-03	9,610644E-04	2,78	0,066118	0,585438
S(A)	21	7,252167E-03	3,453413E-04			
Total (Adjusted)	24	1,013536E-02				
Total	25					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	25	0,32084	5,115333E-02	
A: Groupxxxx				
Day0	8	0,335	6,57021E-03	0,2838467
LIQ/+1	5	0,322	8,310732E-03	0,2708467
LIQ/+4	6	0,3081667	7,586625E-03	0,2570133
LIQ/+6	6	0,3136667	7,586625E-03	0,2625133

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/+4	6	0,3081667	
LIQ/+6	6	0,3136667	
LIQ/+1	5	0,322	
Day0	8	0,335	

27. WHC: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxxxx	3	45,43388	15,14462	3,11	0,066611	0,573514
S(A)	12	58,3797	4,864975			
Total (Adjusted)	15	103,8136				
Total	16					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	16	90,74125		22,68531
A: Groupxxxxx				
Day0	4	93,5375	1,102834	70,85219
S-ICE/-1	4	90,5075	1,102834	67,82219
S-ICE/-4	4	89,7775	1,102834	67,09219
S-ICE/-6	4	89,1425	1,102834	66,45718

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
S-ICE/-6	4	89,1425	
S-ICE/-4	4	89,7775	
S-ICE/-1	4	90,5075	
Day0	4	93,5375	

28. Sensory score: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxxxx	3	61,22461	20,4082	157,30	0,000000*	1,000000
S(A)	28	3,632813	0,1297433			
Total (Adjusted)	31	64,85742				
Total	32					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	32	2,648438	0,3310547	
A: Groupxxxxx				
Day0	8	4,34375	0,1273496	4,012695
S-ICE/-1	8	3,65625	0,1273496	3,325195
S-ICE/-4	8	1,53125	0,1273496	1,200195

S-ICE/-6		8	1,0625	0,1273496
		0,7314453		

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
S-ICE/-6	8	1,0625	S-ICE/-4, S-ICE/-1, Day0
S-ICE/-4	8	1,53125	S-ICE/-6, S-ICE/-1, Day0
S-ICE/-1	8	3,65625	S-ICE/-6, S-ICE/-4, Day0
Day0	8	4,34375	S-ICE/-6, S-ICE/-4, S-ICE/-1

29. Hardness: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power
	(Alpha=0,05)					
A: Groupxxxxx	3	11,72916	3,90972	2,30	0,106552	0,498799
S(A)	21	35,66951	1,698548			
Total (Adjusted)	24	47,39867				
Total	25					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	25	7,4824		1,211704
A: Groupxxxxx				
Day0	8	7,029125	0,4607804	5,817421
S-ICE/-1	6	6,9155	0,5320633	5,703796
S-ICE/-4	5	8,7538	0,5828462	7,542096
S-ICE/-6	6	7,594167	0,5320633	6,382463

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
S-ICE/-1	6	6,9155	
Day0	8	7,029125	
S-ICE/-6	6	7,594167	
S-ICE/-4	5	8,7538	

30. Springiness: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power
	(Alpha=0,05)					
A: Groupxxxxx	3	1,029595E-02	3,431984E-03	1,71	0,196072	0,380698
S(A)	21	4,221101E-02	2,010048E-03			
Total (Adjusted)	24	5,250696E-02				
Total	25					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	25	0,66204		
	0,1054203			
A: Groupxxxxx				
Day0	8	0,689375	1,585106E-02	
	0,5839547			
S-ICE/-1	6	0,6435	1,830322E-02	
	0,5380797			
S-ICE/-4	5	0,6408	2,005018E-02	
	0,5353796			
S-ICE/-6	6	0,6618333	1,830322E-02	0,556413

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
S-ICE/-4	5	0,6408	
S-ICE/-1	6	0,6435	
S-ICE/-6	6	0,6618333	
Day0	8	0,689375	

31. Cohesiveness: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxxxx	3	1,192711E-02	3,975702E-03	10,46	0,000205*	0,994635
S(A)	21	7,985134E-03	3,802445E-04			
Total (Adjusted)	24	1,991224E-02				
Total	25					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	25	0,33248	5,268867E-02	
A: Groupxxxxx				
Day0	8	0,36125	6,894241E-03	0,3085613
S-ICE/-1	6	0,3081667	7,960783E-03	0,255478
S-ICE/-4	5	0,3138	8,720601E-03	0,2611113
S-ICE/-6	6	0,334	7,960783E-03	0,2813113

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
S-ICE/-1	6	0,3081667	Day0
S-ICE/-4	5	0,3138	Day0
S-ICE/-6	6	0,334	Day0
Day0	8	0,36125	S-ICE/-1, S-ICE/-4, S-ICE/-6

32. Resilience: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxxxx	3	9,486627E-03	3,162209E-03	4,64	0,012203*	0,823901
S(A)	21	1,431913E-02	6,818635E-04			
Total (Adjusted)	24	2,380576E-02				
Total	25					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	25	0,31064	4,928533E-02	
A: Groupxxxxx				
Day0	8	0,335	9,232169E-03	0,2857147
S-ICE/-1	6	0,2858333	1,066039E-02	0,236548
S-ICE/-4	5	0,2968	1,167787E-02	0,2475147
S-ICE/-6	6	0,3145	1,066039E-02	0,2652147

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
S-ICE/-1	6	0,2858333	Day0
S-ICE/-4	5	0,2968	Day0
S-ICE/-6	6	0,3145	
Day0	8	0,335	S-ICE/-1, S-ICE/-4

33. WHC: Analysis of Variance Table

Source	Sum of	Mean	Prob	Power

Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxxxxx	3	87,07057	29,02352	10,75	0,001020*	0,987920
S(A)	12	32,38662	2,698885			
Total (Adjusted)	15	119,4572				
Total	16					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	16	90,40437		22,60109
A: Groupxxxxxx				
Day0	4	93,5375	0,8214142	70,93641
LIQ/-1	4	91,5875	0,8214142	68,9864
LIQ/-4	4	89,0325	0,8214142	66,4314
LIQ/-6	4	87,46	0,8214142	64,85891

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/-6	4	87,46	LIQ/-1, Day0
LIQ/-4	4	89,0325	LIQ/-1, Day0
LIQ/-1	4	91,5875	LIQ/-6, LIQ/-4
Day0	4	93,5375	LIQ/-6, LIQ/-4

34. Sensory score: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxxxxx	3	16,75586	5,585287	19,08	0,000001*	0,999996
S(A)	28	8,195313	0,2926897			
Total (Adjusted)	31	24,95117				
Total	32					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	32	3,664063	0,4580078	
A: Groupxxxxxx				
Day0	8	4,34375	0,1912752	3,885742
LIQ/-1	8	4,28125	0,1912752	3,823242
LIQ/-4	8	3,46875	0,1912752	3,010742
LIQ/-6	8	2,5625	0,1912752	2,104492

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/-6	8	2,5625	LIQ/-4, LIQ/-1, Day0
LIQ/-4	8	3,46875	LIQ/-6, LIQ/-1, Day0
LIQ/-1	8	4,28125	LIQ/-6, LIQ/-4
Day0	8	4,34375	LIQ/-6, LIQ/-4

35. Hardness: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxxxxx	3	6,048713	2,016238	0,75	0,536361	0,180822
S(A)	20	53,92981	2,696491			
Total (Adjusted)	23	59,97852				
Total	24					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	24	7,215		1,199991
A: Groupxxxxxx				
Day0	8	7,029125	0,5805698	5,829134
LIQ/-1	6	8,073667	0,6703843	6,873675

LIQ/-4	5	6,8256	0,7343692	5,625608
LIQ/-6	5	6,8714	0,7343692	5,671409

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/-4	5	6,8256	
LIQ/-6	5	6,8714	
Day0	8	7,029125	
LIQ/-1	6	8,073667	

36. Springiness: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxxxxx	3	3,35215E-03	1,117383E-03	0,74	0,538245	0,180146
S(A)	20	3,002547E-02	1,501274E-03			
Total (Adjusted)	23	3,337763E-02				
Total	24					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	24	0,673625	0,1118906	
A: Groupxxxxxx				
Day0	8	0,689375	1,369888E-02	0,5774844
LIQ/-1	6	0,672	0,0158181	0,5601094
LIQ/-4	5	0,6618	1,732786E-02	0,5499094
LIQ/-6	5	0,6622	1,732786E-02	0,5503094

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/-4	5	0,6618	
LIQ/-6	5	0,6622	
LIQ/-1	6	0,672	
Day0	8	0,689375	

37. Cohesiveness: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxxxxx	3	1,129356E-02	3,764519E-03	12,04	0,000101*	0,998023
S(A)	20	0,0062534	3,1267E-04			
Total (Adjusted)	23	1,754696E-02				
Total	24					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	24	0,3367083	5,544792E-02	
A: Groupxxxxxx				
Day0	8	0,36125	0,0062517	0,3058021
LIQ/-1	6	0,3435	7,218841E-03	0,2880521
LIQ/-4	5	0,3054	7,907844E-03	0,2499521
LIQ/-6	5	0,3206	7,907844E-03	0,2651521

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/-4	5	0,3054	LIQ/-1, Day0
LIQ/-6	5	0,3206	LIQ/-1, Day0
LIQ/-1	6	0,3435	LIQ/-4, LIQ/-6
Day0	8	0,36125	LIQ/-4, LIQ/-6

38. Resilience: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Groupxxxxxx	3	0,0141613	4,720433E-03	12,19	0,000093*	0,998223
S(A)	20	7,742033E-03	3,871017E-04			
Total (Adjusted)	23	2,190333E-02				
Total	24					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	24	0,3148333	5,180139E-02	
A: Groupxxxxxx				
Day0	8	0,335	6,956127E-03	0,2831986
LIQ/-1	6	0,3348333	8,032244E-03	0,2830319
LIQ/-4	5	0,279	8,798882E-03	0,2271986
LIQ/-6	5	0,2944	8,798882E-03	0,2425986

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/-4	5	0,279	LIQ/-1, Day0
LIQ/-6	5	0,2944	LIQ/-1, Day0
LIQ/-1	6	0,3348333	LIQ/-4, LIQ/-6
Day0	8	0,335	LIQ/-4, LIQ/-6

39. Hardness: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Group	3	9,63923	3,213077	0,84	0,488271	0,196269
S(A)	18	68,63681	3,813156			
Total (Adjusted)	21	78,27604				
Total	22					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	22	7,575227		1,37878
A: Group				
LIQ/-1	6	8,073667	0,7971989	6,694886
S-ICE/-1	6	6,9155	0,7971989	5,53672
ICE/+1	5	6,9342	0,8732876	5,55542
LIQ/+1	5	8,4098	0,8732876	7,03102

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
S-ICE/-1	6	6,9155	
ICE/+1	5	6,9342	
LIQ/-1	6	8,073667	
LIQ/+1	5	8,4098	

40. Springiness: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Group	3	8,482873E-03	2,827624E-03	2,06	0,141994	0,439110
S(A)	18	0,0247539	1,375217E-03			
Total (Adjusted)	21	3,323677E-02				
Total	22					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	22	0,6433182	0,1167045	
A: Group				
LIQ/-1	6	0,672	1,513944E-02	0,5552955
S-ICE/-1	6	0,6435	1,513944E-02	0,5267954
ICE/+1	5	0,6334	1,658443E-02	0,5166954
LIQ/+1	5	0,6186	1,658443E-02	0,5018954

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
LIQ/+1	5	0,6186	
ICE/+1	5	0,6334	
S-ICE/-1	6	0,6435	
LIQ/-1	6	0,672	

41. Cohesiveness: Analysis of Variance Table

Source Term	DF	Sum of Squares	Mean Square	F-Ratio	Prob Level	Power (Alpha=0,05)
A: Group	3	4,309821E-03	1,436607E-03	2,83	0,067395	0,579530
S(A)	18	9,125133E-03	5,069518E-04			
Total (Adjusted)	21	1,343495E-02				
Total	22					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	22	0,3300455	6,008485E-02	
A: Group				
LIQ/-1	6	0,3435	9,191952E-03	0,2834151
S-ICE/-1	6	0,3081667	9,191952E-03	0,2480818
ICE/+1	5	0,332	1,006928E-02	0,2719151
LIQ/+1	5	0,3382	1,006928E-02	0,2781152

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
S-ICE/-1	6	0,3081667	
ICE/+1	5	0,332	
LIQ/+1	5	0,3382	
LIQ/-1	6	0,3435	

42. Resilience: Analysis of Variance Table

Source	Sum of	Mean	Prob	Power

Term	DF	Squares	Square	F-Ratio	Level	(Alpha=0,05)
A: Group	3	7,839906E-03	2,613302E-03	2,94	0,061190	0,596667
S(A)	18	1,600887E-02	8,893815E-04			
Total (Adjusted)	21	2,384877E-02				
Total	22					

Means and Effects Section

Term	Count	Mean	Standard Error	Effect
All	22	0,3123182	5,682121E-02	
A: Group				
LIQ/-1	6	0,3348333	1,217498E-02	0,2780121
S-ICE/-1	6	0,2858333	1,217498E-02	0,2290121
ICE/+1	5	0,3074	1,333703E-02	0,2505788
LIQ/+1	5	0,322	1,333703E-02	0,2651788

Duncan's Multiple-Comparison Test

Group	Count	Mean	Different From Groups
S-ICE/-1	6	0,2858333	
ICE/+1	5	0,3074	
LIQ/+1	5	0,322	
LIQ/-1	6	0,3348333	