# **PROGRESS REPORT**

1<sup>st</sup> Quarter (November 2023 – February 2024)

# PROGRAM TITLE:

Accelerating Salt Research and Innovation Center (ASIN Center)

# **PROJECT 4 TITLE:**

## Value-Adding of Sea Salt and Utilization of its Byproducts



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#### SUMMARY SHEET

**1) Program Title:** Accelerated R&D Program for Capacity Building of Research and Development Institutions and Industrial Competitiveness: Niche Centers in the Regions for R&D (NICER): Accelerating Salt Research and Innovation (ASIN Center)

**Project Title:** Value-adding of Sea Salt and Utilization of its Byproducts

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(2) Cooperating Agencies DOST-PCIEERD, DOST-Region 1, PSU-FIC, MMSU

(3) Site/s of Implementation (Barangay / Municipality / District / Province / Region / Country)

Base Station: DMMMSU-NLUC-FRTI, Paraoir, Balaoan, La Union Site/s of Implementation: DMMMSU - College of Fisheries, Sto. Tomas, La Union MMSU – College of Engineering, Batac, Ilocos Norte

(4) Project Duration November 2023 – November 2025

(7) Total Budget

AGENCY/ PARTNER	PS	MOOE	СО	Sub-Total
DOST	1,482,156.00	4,655,480.00	1,860,000.00	7,997,184.00
DMMMSU		2,680,000.00		2,680,000.00
TOTAL				10,677,184.00

#### INTRODUCTION

Sea salt at different production methods will be used in this project. Value-adding sea salt will be done to improve its quality and acceptability to consumers. This will allow the Region 1 to expand their products to different food industries/sector. This project wants to utilize the sea salt produced at different production method to standardize its application in the development gourmet/flavored salt, utilize its byproducts and use in pottery as glazing material

While it is true that some products are more valuable in their raw state, there is evidence to show that more benefits accrue from transforming primary products into finished goods. Higher prices and increased profits can be realized from the sale of value-added goods. In addition, value added commodity are able to stand out and be differentiated from competitor products through branding standardized processing, and improved packaging. In the long run value addition also results in cost efficiency as manufacturers are able to produce higher value goods with a given set of resources.

Through value addition of sea salt and development of products from its byproduct/waste can be addressed which can be curtailed to reduce postharvest losses by 12-15%. Value adding of sea salt will be done by incorporating dehydrated fish flesh which can be used as flavored salt for cooking. Byproduct like nigari liquid (bitter liquid/ magnesium chloride) will be collected and characterize to evaluate its use for food applications. To fully utilize the use of salt to non-food product, salt will be used to glaze the pots to enhance the color, presentation and strength of the product.

#### OBJECTIVES

## General:

Develop gourmet/specialty sea salt from different production methods, standardize and characterize its properties for human consumption, and utilize salt by-products for other uses.

#### Specific:

- 1. Develop, standardize and characterize flavored salts
- 2. Collect and evaluate/characterize byproducts from sea salt produced at different methods
- 3. Develop salt glazing for clay products using sea salt

#### **REVIEW OF RELATED LITERATURE**

#### Salt Production and Consumption World-Wide

Recently, the annual world production of salt has exceeded 250 million tons. Approximately one third of the total is produced by solar evaporation of sea water or inland brines. Another third is obtained by mining of rock salt deposits, both underground and on the surface. The balance is obtained as brines, mainly by solution mining. Brines can be used directly (for example in diaphragm electrolysis) or thermally evaporated to produce vacuum salt.

The purity of washed solar salt produced in India and China reach 99 - 99.5% (NaCl, dry bases) but solar salt produced in Australia and Mexico is 99.7 – 99.8% pure. The purity of processed rock salt fluctuates between 97 and 99%+ in the USA and in Europe. Vacuum salt is usually 99.8 - 99.95% pure (Ahmmed & Mutwkel, 2019).

The chemical industry is the largest salt consumer of salt using about 60% of the total production. This industry converts the salt mainly into chlorine, caustic soda and soda ash without which petroleum refining, petrochemistry, organic synthesis, glass production, etc. would be unthinkable.

The second largest user of salt is mankind itself. Humans need about 30% of the total salt produced to support their physiological functions and eating habits. Salt for food is the most "taken for granted" commodity, available from thousands of sources in hundreds of qualities as table, cooking and salt for food production. About 10% of salt is needed for road de-icing, water treatment, production of cooling brines and many other, smaller applications. Whatever the use of salt, it is the sodium chloride in the salt that is required and not the impurities.

#### Methods of Salt Production

#### Solar Evaporation Method in Ponds and Geomembrane

This is the oldest method of salt production. It has been used since salt crystals were first noticed in trapped pools of sea water. Its use is practical only in warm climates where the evaporation rate exceeds the precipitation rate, either annually or for extended periods, and ideally, where there are steady prevailing winds. Solar salt production is, typically, the capturing of salt water in shallow ponds where the sun evaporates most of the water. The concentrated brine precipitates the salt which is then gathered by mechanical harvesting machines. Any impurities that may be present in the brine are drained off and discarded prior to harvesting (Rathnayaka et al. 2014).

Usually two types of ponds are used. First is the concentrating pond, where the salty water from the ocean or salt lake is concentrated. The second is called the crystallizing pond, where the salt is actually produced. Crystallizing ponds range from to 40 to 200 acres with a foot-thick floor of salt resulting from years of depositions. During the salt-making season of four to five months, brine flows continuously through these ponds. This is a saturated brine solution, containing as much salt as it can hold, so pure salt crystallizes out of the solution as the water evaporates. Natural chemical impurities are returned to the salt water source.

High-density polyethylene (HDPE) geomembranes have been commonly used in composite liners at the bottom of modern municipal solid waste landfills, and they have shown excellent performance in containing a broad range of chemicals (Rowe and Yu, 2019; Jumaeri et al. 2004). Due to the advantages of HDPE geomembranes, then in this study will be applied to produce salt with high purity. Various methods of high purity salt production have been exploited by using binder agent impurity. The use of HDPE geomembranes was introduced in the manufacture of super quality salt. Also, the removal of impurities by using Na<sub>2</sub>CO<sub>3</sub> and NaOH (Bahruddin et al. 2003; Choi et al. 2016), as well as Na<sub>2</sub>CO<sub>3</sub> and Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> in high-quality salt production also reported. Although the method can improve the quality of salt produced, the method is not appropriate when applied and industrial scale, because of not only high production costs but also not environmentally friendly. Therefore, it is necessary to develop alternative methods that can solve the problem. In a previous study, using black plastic as a liner in the manufacture of salt can be produced the high-quality salt (Kumar et al. 2009).

#### Vacuum Evaporation Method

Another method of salt production is the evaporation of salt brine by steam heat in large commercial evaporators, called vacuum pans. This method yields a very high purity salt, fine in texture, and principally used in those applications requiring the highest quality salt.

The first part of the operation is known as solution mining. Wells are drilled from several hundred to 1,000 feet apart into the salt deposit. These wells are connected via lateral drilling, a recently developed technology. Once the wells are connected, the solution mining operation begins: water is pumped down one well, the salt below is dissolved, and the resulting brine is forced to the surface through the other well. It is then piped into large tanks for storage (Rathnayaka et al. 2014).

Next, the brine is pumped into vacuum pans. These are huge closed vessels under vacuum about three stories high. They are normally arranged in a series of three, four or five, with each one in the line under greater vacuum than the preceding one. This series of vacuum pans operates on a very simple principle: Whenever pressure is lowered, the temperature at which water will boil is also lowered. For instance, under normal air pressure at sea level, water boils at 212°F. But at ten thousand feet above sea level, where air pressure is much less, water boils at 194°F. Vacuum pans may operate at as low as 100°F.

In the vacuum pan process, steam is fed to the first pan. This causes the brine in the pan to boil. The steam from the boiling brine is then used to heat the brine in the second pan. The pressure in the second pan is lower, allowing the steam made by the boiling in the first pan to boil the brine in the second pan. The pressure is reduced still further in each succeeding pan. This allows the steam made by the boiling brine in the previous pan to boil the brine in the next pan. While the boiling operation could be done with just one pan, several pans in a row produce more salt per pound of steam, thus allowing greater energy efficiency.

#### Researches Related to the use of Plastic Materials in Salt Production

Aypa (1977) studied the viability of salt-making in polyethylene plastic material as a small-scale industry in the Philippines. According to the researcher, to support the different needs for salt in the country, it became necessary to produce the maximum amount of salt with the least capital investment. This problem can be resolved only if an economical material for salt production can be found and many people can be encouraged to go into business. The study uses compartment measuring 1m x 8 x 2 inch in which 4-5 gantas of salt produced daily if the sun is hot and the stock water placed has a salinity of  $200^{\circ}/_{oo}$  or higher. Based on the results of the study, the intensity of the solar heat and weather condition has the greatest effect on the evaporation of seawater resulting in the formation of salt. Salt formation further enhanced by the ground temperature. It was observed that when the atmospheric temperature ranges from 26°C - 29°C, and the ground temperature ranges from  $40^{\circ}$ C -  $52^{\circ}$ C, a daily evaporation of 12-15% of seawater contained in a plastic compartment with a surface area of 6 sq.ft and with a depth of 2 inches can be obtained.

Quality monitoring of salt produced in Indonesia through seawater evaporation in HDPE geomembrane lined ponds as conducted by Jumaeri et al. 2018. HDPE geomembrane is used to coat evaporation ponds with viscosity 12-22° Be and crystallization ponds with a viscosity of 23° Be. The monitoring of the product is carried out in the particular periods during the salt production period. The result of control shows that the quality of salt produced in HDPE geomembrane coated salt ponds has an average NaCl content of 95.75%, so it has fulfilled with Indonesia National Standard (SNI), that is NaCl > 94.70%. Based on the results of the study, production of salt with HDPE geomembrane can improve the quality of salt product from NaCl 85.4% (conventional system) to 95.75%.

High-density polyethylene (HDPE) geomembranes have been commonly used in composite liners at the bottom of modern municipal solid waste landfills, and they have shown excellent performance in containing a broad range of chemicals (Rowe et al. 2016; Booker et al. 2004). Due to the advantages of HDPE geomembranes, then in this study will be applied to produce

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#### Value-added Sea Salt Products

#### Gourmet Salt

Gourmet salt is a naturally harvested raw sea salt (primarily sea salt) that adds flavor and dressing to food. It also gives food an almost exotic flavor and moisture. It is high-quality salt commonly used in cooking to improve the taste and attractiveness of food. Gourmet salt has a high mineral content and better solubility. It can be added to different types of spices and herbs to improve the aroma and color of the fragrance. Gourmet salt is not refined, but it is harvested naturally with relatively low sodium content and without additives. As exposure to global media and international trade rises, the awareness for a variety of dishes among consumers has risen. By using gourmet salt, chefs enhance their recipes by improvising and enhancing their sensual sensations, increasing their appeal. Gourmet salt is used to preserve canned foods to extend shelf life. Fleur de sel is one of the most popular gourmet salts used in a variety of foods such as grilled meat, vegetable dishes and salads. Gourmet salt is also used to keep food longer to preserve canned food.

The global Gourmet salt market size was calculated to be USD 8 billion in 2022, and it is envisioned to reach USD 13 billion by the end of 2028 at a CAGR of 6.3% over the prediction time. The rapid urbanization of emerging economies and the changing lifestyle of consumers have increased the call for gourmet salt.

Europe was the largest consumer and manufacturer of salt products due to demand for processed foods in European countries, including Germany, France, Spain and Italy. Western Europe is considered as the most abundant and fastest-growing market for gourmet salt. It is the largest producer and consumer of gourmet salt. The high demand for processed foods is driving the market mainly in countries such as Italy, Germany, Spain and France. North America is anticipated to witness growth in the near future due to the preferences of American consumers and strict regulatory changes in food. Key factors driving the market in the Asia Pacific region include better living standards, health-conscious behavior, emerging economies and the food and beverage industry. Asia-Pacific is foreseen to grow significantly in the coming years due to emerging economies, rising standards, an increase in demand for processed foods, and advances in the food and beverage industry, driven by health-conscious behavior. The gourmet salt market segmentation analyses are categorized based on type, application and

geographical. The common types of gourmet available in the market are the following with brief description:

## a. Himalayan Pink Salt (The king of Salt)

Himalayan Pink Salt is the purest and arguably the most majestic of all the salts. Many consider it the King of Salts. It is an unrefined, unprocessed raw mineral mined from caves formed 250 million years ago in the Himalayan mountain range. The salt from these mines has experienced tremendous pressure over the years, and for this reason is extremely pure. It is also packed with trace minerals like iron, potassium, magnesium and calcium, which are responsible for its gorgeous pink hue and its bold, intense flavor. It is fantastic for brining or for seasoning

something with a subtle flavor, such as lean meats or steamed vegetables - a delicious alternative to your standard table salt.

## b. Fleur de Sel (The Queen of Salts)

Considered by many as the Queen of Salts, Fleur de Sel is a salt that is harvested using traditional Celtic methods in Guerande on the western coast of France. This exceptional salt is hand-harvested according to Celtic tradition and is characterized by irregular, relatively fine crystals. The smaller crystals dissolve quickly, while the larger crystals take longer, and as such, the delicate salt flavor unravels across your palate in a wave. Regarded by many chefs as the finest of all sea salts, it is favored for its elegant appearance, complex flavour and moist texture. It is suited to all foods - from fried eggs to veggies, salads and roasted or baked meats or fish.

#### c. All-Rounders Salts

#### Kosher Salt

Kosher salt is an excellent all-rounder, and should be a key weapon in the arsenal of any chef. Its flakey texture makes it easy to pinch when cooking. This means you can distribute your salt with greater precision. It is also less refined than table salt, and thus more pure. The flakes aren't quite as compact as table salt, meaning it isn't as dense The larger flakes distribute evenly and take longer to dissolve, which makes it perfect for seasoning meat or veggies with your hands. Kosher is the salt of choice for pickling and brining because it has no added anticaking agents, which can turn the liquid brown. It is also the salt of choice for smoking as the smoke can penetrate the layers of the flakes, infusing all that smoky goodness. But don't stop there! Kosher salt is perfect for anything that calls for "coarse salt", and is a fantastic alternative to your standard table salt.

#### Sel Gris

Sel gris is another excellent all-rounder. It comes from the same salt pans as Fleur de Sel, but is harvested using different techniques. The striking grey color comes from the French clay lining the ponds. This delectable all-rounder brings depth to food when dissolved in cooking, has moist crystals which don't dehydrate meat, and the coarse grain variety has a hearty crunch when used as a finishing salt.

#### d. Black Salts

#### Indian Black Salt

Also called Kala Namak, Indian black salt is a little deceiving; it is actually pinkish brown in colour. Made from crushed Indian volcanic rock salt, it is unrefined and full of minerals (such as sulfur, which imparts an intriguing aroma). It is packed full of flavor too, and is perfect for a range of Indian dishes like pickles, salads, chutneys, curries and raitas.

#### Cyprus Black Flake Salt

Cyprus black flake salt is a Mediterranean sea salt that naturally forms in beautifully large pyramid-shaped crystals that have been blended with activated charcoal to give it a dramatic black color. It is a bold and powerful salt with a unique earthy flavor. Perfect for seasoning on pretzels or on bread, on fish, roast veggies, a caprese salad, frittata, haloumi, tomatoes and over fruit.

#### Hawaiian Black Lava Salt

Hawaiian Black Lava salt is a naturally derived Hawaiian sea salt that has been blended with activated charcoal derived coconut shells. This black wonder is at its best when paired with red meat, pork, fish, eggplant, potatoes, sashimi, scallops, fruit, kale, buttered popcorn and avocado.

#### e. Smoked Salts

Smoky salts are an intriguing bunch, and are amongst the boldest, most exciting ways you can season your dishes. Hand-crafted using a slow smoking process with real wood, they are an excellent way to add a smoky complexity to your dishes without resorting to the artificial bitterness of liquid smoke.

## f. Chilli Salts

Chilli salts are crafted by fusing the flavour of naturally spicy ingredients to sea salt crystals, and are a brilliant way to add a fiery kick to any dish. Ranging from subtle to explosive, chilli salts can take a dish to a whole new level. They are excellent for seasoning food that might be lacking in complexity or zing.

#### g. Flavored Salts

Flavored salts are those that have been naturally infused with another natural flavor companion, creating something that is so much more than just a seasoning. No blends, powders or extracts are used; all flavored salts are infused with fresh, natural ingredients like herbs, vegetables and fruits. They can turn any old dish into a flavored-packed, mouth-watering delight.

The Department of Science and Technology – Industrial Technology and Development Institute (DOST-ITDI) developed gourmet salts infused with extracts of seaweed and sea grapes, shiitake mushroom, shrimp heads, guava, mango, and tamarind leaves which offer authentic delicacy and saltiness to any dish and concoction. The technology aims to give variations to salt taste and add richness to its flavor. Using local ingredients, these improved salt products are expected to add value to locally-produced salt.

#### **By-products from Sea Salt Production**

Sodium chloride, NaCl (halite), the most common evaporite salt, is used in several forms by virtually every person in the world. Most people think that the most common usage area of salt is related to food processing, or, in colder climes, to road deicing. In fact these are lesser usages of halite; it is mostly used as feedstock in the chemical industry. There are more than 14,000 reported usages of halite, and it, along with other salts, has long played a very important role in human affairs. A major portion was devoted to a discussion of more than 40 kinds of evaporite salts, such as thenardite, bloedite, mirabilite, carnalite, langbeinite, bischofite and kieserite etc. from brine and hoe to recover it in usable form. Potassium chloride salt is used mostly as agricultural fertilizer; while the magnesium sulphate salt is chiefly used for refractory materials, packaging and transportation in the formation of a wide range of industrial applications. Magnesium chloride salts are used in metal production, textile, paper, ceramic, and cement and sodium sulphate salts are utilized in detergent powder, glass, pulp and paper (Hardie, 1991; Harben, 1990; Warren, 1999).

Salt exists as a solution in the seas, lakes and salty water sources; whereas it is found as a solid in the form of rock-salt. It is not only mined, but it has also been produced more economically by solar concentration of brines for centuries. The salts formed at the more saline end of a phreatic precipitation sequence make up of progressively more soluble evaporitic minerals: silicates (zeolites), calcite, Ca– , Na– , (K) and Mg-sulphates, chromates, borates and perchlorates. Two specific precipitation trends can be distinguished within this more general regional sequence. MgNa-(Ca)-SO<sub>4</sub> -Cl type brine is characterized by an association of bischofite, bloedite, epsomite, glauberite, gypsum, halite, hexahydrite, kieserite, and a Ca-Mg-Na-(K)-Cl type brine related to antarcticite, bischofite, carnalite, halite, sylvite (Lehrman, 1978). In the natural systems, the secondary salts, such as sylvite, mirabilite, thenardite, bloedite and polyhalite, form syndepositionally via interaction between highly evolved near surface brines and earlier formed minerals (McCaffrey et al., 1987). Sodium sulphate salts (salt cake or Glauber's salt) are relatively common salt components precipitated in many saline lakes and playas. Thenardite and mirabilite — the anhydrous and decahydrate single sodium sulphate salts — are commercially important, as are glauberite and bloedite. Thenardite is extremely

hygroscopic and a colourless to white mineral forming the decahydrate mirabilite. Natural potash evaporites are part of the bittern series precipitated at the surface or in the shallow subsurface at the higher concentration end of the evaporation series. Commercial potash can be potassium chloride (KCI), potassium sulphate (K2 SO4 ), potassium-magnesium sulphate (K2 SO4 .MgSO4 ), potassium nitrate. The most common naturally occurring potash minerals are carnalite, sylvite and langbeinite, with sylvite the most economically important.

## **Ceramic Glaze**

As glazes became more sophisticated, a flux was added to the ground-glass and water mixture to lower the melting temperature of the glass. The flux also affected the properties and appearance of the glaze. An alkaline glaze made with a flux of ash (containing soda ash from burned marine plants or potash from the ash of forest plants) is transparent, as is a glaze fluxed with lead (usually "red" lead or lead tetroxide). Glazes fluxed with tin oxide, however, are opaque and white and are similar to a layer of paint. Glazes can be colored via the addition of metallic oxides: cobalt produces grayish to bright blue; manganese, bright red- purple to purplish brown; antimony, yel- low; copper, a blue, green, or bluish red; and iron, from pale yellow, to orange- red, to black. Uranium has been used to give brilliant shades of orange and yel- low. The composition and uses of new kinds of glazes excited keen interest among potters. In the Near East, many potters kept and exchanged meticulous records of their experiments with differ- ent additives. In China, the art of glazing took a dif- ferent turn. Chinese pottery from the Sung Dynasty (960-1279 A.D.) was covered with a glaze made of ground feldspar, a verifiable silicate mineral later used in the manufacture of porcelain. Chinese potters also occasionally used a lead flux (lead tetroxide) in their glazes, but most of their stoneware was salt-glazed.

In the salt-glazing process, a shovelful of common salt (NaCl) was thrown into the kiln at its hottest point. The heat dissociates the salt into its sodium and chlorine components; the chlorine gas escaped out the chimney, but the sodium combined with silica occurring naturally in the clay, resulting in a smear glaze of sodium-silicate over the body of the item. A salt-glazed pot has a pitted texture, like the peel of an orange. Adding a small amount of red lead to the shovelful of salt resulted in a smoother glaze. The Assyrians first used glaze (possibly as early as 1100 B.C.) to cover deco- rated bricks. Opaque white tin glaze was probably first used to mask blemishes and discolorations in the main body of an object. Most clay contains variable amounts of iron oxide that can change the color of baked clay in splotches from dark red to light buff.) Panels made of glazed brick have been found in the ruins of Khorsabad, Susa, Nimrud, and Babylon (Anderson, K.J., 2013).

## SCIENTIFIC BASIS/THEORETICAL FRAMEWORK

Value adding of sea salt and utilization of its byproduct is important because it encourages customers to choose the product by giving them more. This could be as a desirable product/ brand, or unique product features. A product might sell for the price that a customer is willing to pay and their perception of how much the product is worth.

Needs	Solution
<ul> <li>The Philippines is missing out on potential export market for salt</li> <li>Increasing demand for gourmet salt export in Asia and European countries</li> <li>Potential of by-products from sea salt production to develop other products for food industry</li> <li>Potential of locally produced sea salt to be used in non-food industry in the Philippines as another line of market and opportunities</li> </ul>	<ul> <li>Add value to the regular salt/sea salt that can be used by food industry (flakes salt and fish flavored salt)</li> <li>Utilized the by-products from salt production to create new product and market opportunity</li> <li>Explore the other use of locally produced salt to non-food industry to create new opportunities</li> </ul>
Differentiation	Benefits/Advantage
<ul> <li>Standardized procedure on the development of flakes salt and fish flavored salt supported by the laboratory testing and statistical analysis</li> <li>By-products are produced locally at different production methods</li> <li>New form of value-addition in regular/locally produced salts from the products developed by DOST-ITDI and commercially available in the market (particularly in fish flavored salt that utilized the fish/commodity locally produced in the Region)</li> </ul>	<ul> <li>Additional product, livelihood, New market opportunity for fishery and salt industry</li> <li>Products that could compete nationwide and globally</li> <li>Additional trademark for value-added salts</li> </ul>

Figure 1. Framework of the Project

#### METHODOLOGY

#### Study 1 Title: Development and Characterization Gourmet Sea Salt (Flaked Salt and fish flavored salt)

#### A. Benchmarking to Japan and India

Before the start of the development of gourmet sea salt, a visit to the salt center in Japan and India will be done to gain additional knowledge/skills on how they process salt for human consumption. Observation, interview and hands-on training (if possible) in their salt processing plant/area will help the proposed project to implement successfully. It is expected that after the benchmarking, innovative technologies and processes on salt processing/ value-adding from the two countries will enhance the proposed gourmet products in this study (the composition and process).

For the utilization of salt by-products, the benchmarking in Japan will be very helpful for the development of Nigari liquid to obtain improvement on standardization of the product (from collection, processing and laboratory testing). Japan is the country who first developed the Nigari liquid extracted from the saltern ponds. Exposure to their Salt Center will help the research team to obtain clear picture on the post-harvest/handling, processing and marketing of the product based on the demand and uses of the product.

## B. Raw Materials and Experimental Design for Gourmet Sea Salt Production

Sea salt produced from different production methods at the DMMMSU-NLUC-FRTI, Paraoir, Balaoan, La Union will be used in the development of gourmet salt. Development of gourmet/flavored salt will be conducted/ implemented by the DMMMSU in La Union and PSU-FIC in Pangasinan.

Two (2) gourmet products are target output of this study which includes the salt flakes, and fish flavored sea salt. All salts will be cleaned, washed and dried (at least 5% moisture content obtained) to ensure that it will not affect the quality of the proposed products, and safe to eat. Cooking facility, salt washer and spin dyer developed by the DOST-ITDI will be used to clean and enhance the quality of salt before the processing/ value-adding. All gourmet salt products will be subjected at different treatments with three replicates or trials. Treatments will be based on the formulation (ratio of ingredients and methods of production). Mixture design will be used to optimize formulation, while factorial design and response surface methods (RSM) will be used to optimize the process.

## C. Methods on Gourmet Salt Production

#### C.1 Development of Flavored Salt Flakes

Flake salt is a common category of salt distinguished by the unique, flattened shape of flaked salt crystals. Flake salts are generally created by mechanically compressing standard granulated salt crystals, which are originally cubic in shape. These new, flat crystals have a larger surface area and lower bulk density than granulated salt, which enables it to perform differently in food applications. Compared to granulated salt, flake salts will stick better to food, dissolve faster, and blend more evenly.

## Flake salts in this study will be prepared by the following procedures:

1. Sea salt will be dissolved in distilled water at different ratio with herbs/spices. The combined salt and water will be heated in a hot plate at different temperature (60, 70, 80, 90, 100 °C) and drying methods which will be assigned as treatments (with three replicates) of the study (see

table 1). Herbs that will be added to the mixture are Rosemary, Thyme, Oregano, Basil, Parsley, while dried spices are Garlic powder, Onion powder, Chili seasoning.

2. Electric induction cooker/heater will be used to control the temperature, while ceramic plates/pan will be vessel of the salt and water mixture during heating to prevent corrosion/rusting. The heating of the mixture will be continued until thin and crusty layer of salt appeared. The time that the crusty layer of salt appeared will be recorded.

3. The produced flaky salts at different treatments will be vacuum packed samples (1 kg per replicate) to be submitted to the DOST-ITDI or ADMATEL for physical and chemical analysis. Other options are laboratory accredited by the government (3<sup>rd</sup> party) applicable to the target parameters.

Γ					-	
Treatment	Drying	Yield	Physical	Chemical	Sensory	Gas
	time (hrs.)	(g)	Properties	Properties	Evaluation	Chromato
	( ,	(0)	(FTIR and			graphy
			SEM)			3
Sun drying			02)			
(control)						
60 °C (hot plate)						
70 °C (hot plate)						
· · · · ·						
80 °C (hot plate)						
90 °C (hot plate)						
,						
100 °C (hot plate)						
Solar tunnel dryer						
Dehydrator						
<b>,</b>						

 Table 1. Heating of mixture at different temperature/drying methods

## C.3 Development of Fish Flavored Sea Salt

The sea salt will be flavored with dehydrated and powdered flesh/meat of milkfish and tilapia. Milklfish was selected species/commodity in this study since Region 1 specifically the Pangasinan and La Union, is one of the top producer of Milkfish in the country. Moreover, species of tilapia (Blackchin, *Sarotherodon melanotheron*) is also abundant in Lingayen gulf, Sto. Tomas Cove and other parts of the coastal waters in Region 1. Blackchin tilapia is considered as pest to fish cages and fish pen farmers/ operators in Region 1 which makes it potential commodity to be utilized in this study.

Three kilos (3 kg) of sea salt will be used in each type of flavoring. The treatments for the mixture of milkfish meat/flesh, Tilapia and salt will be replicated thrice. Milkfish and blackchin tilapia will be fillet/removed, weighed, dehydrated and pulverized. Sea salt, herbs/spices and powdered milkfish and blackchin tilapia will be mixed at different ratio and heated in pan on low flame for 5 minutes until the salt begins to change color and the aroma of fish begins to rise. Different ratio of sea salt and milkfish will be assigned as treatments of the study (table 2). Developed products will be packed and store for nutrient/chemical analysis and sensory evaluation. Herbs that will be added to the mixture are Rosemary, Thyme, Oregano, Basil, Parsley, while dried spices are Garlic powder, Onion powder, Chili seasoning.

Samples (at least 1 kg per replicate) will be submitted to the DOST-FNRI, DOST-ITDI or ADMATEL for physical and chemical analysis. Other options are laboratory accredited by the government (3<sup>rd</sup> party) applicable to the target parameters.

## Table 2. Treatments for fish flavored salt

Treatment	Physical Properties (FTIR and SEM)	Chemical/Nutrient Analysis (NaCl, Protein, Magnesium, Calcium etc)	Sensory Evaluation	Gas Chromato graphy	Shelf life testing	
Salt+Herbs/S pices: Milkfish at different mixture						
Salt+Herbs/S pices: Tilapia at different mixture						

## D. Acceptability of Flake salts and Fish Flavored Sea Salt using Sensory Evaluation

## D.1 Descriptive Attribute Rating Test

After formulation, samples will be subjected to Descriptive Attribute Rating Test to determine the appropriate attribute using the work of Amerine et.al 1965 and their level of intensities. This test will be done using a structured scale from the work of Meilgaard et al 1991) with a rating of 0=None, 1= Threshold, 2= Very Slight, 3 = Slight, 4= Slight -Moderate 5= moderate, 6= moderate-strong and 7= strong. In conducting the test, a total of 10 panelists will be needed during evaluation; each panelist will be provided with coded samples at least 28 grams per treatment (Sales, 2019). Samples will be presented to the panelists and they will be provided with spitting cups and distilled water.

## D.2 Acceptability Test

After descriptive attributes test, products will be subjected to acceptability test. A total of 100 untrained panelists will be selected during the study. During testing, consumers will be provided with 56 g of samples for

three trials using metallic plate which will be coded and presented one at a time. Coding and presentation of samples in different order will be done using table of random numbers to guard against any bias (Steel, 1960). Likewise, consumers will be provided with score sheet utilizing Hedonic Scale (Jones et.al, 1955) with the rating of 9= like extremely, 8= like very much, 7=like moderately, 6= like slightly, 5= neither like nor dislike, 4 = dislike slightly, 3= dislike moderately, 2= dislike very much and 1= dislike extremely. Products will be evaluated three times based on the attributes identified during the descriptive evaluation.

# E. Fourier-Transform Infrared (FTIR) Spectroscopy and Scanning Electron Microscopy (SEM)

Salt samples will be sent to DOST-ADMATEL for FTIR and SEM analysis. FTIR will be conducted to identify and verify the chemical bonds/composition of sea salt. SEM will be conducted to determine traces of debris and contaminants in sea salt. To comply with the codex standards for food grade salt, chemical, microbial and heavy metal analyses will be done. For these analyses, flaked salt samples will be sent to DOST-ITDI to test the moisture, NaCl content, insoluble matter, sulfate, Iodine, Ca, Mg, K, Cu, , microbial content, heavy metals (As, Hg, Pb, Cd), and histamine for flavored salt.

## F. Statistical Analysis

All the collected data will be subjected to statistical analysis with ANOVA and any difference between the treatments means will be performed by Duncan Multiple Test using SPSS version 23.

#### Study 2: Product Development from Sea Salt By-product

#### A. Collection and Characterization of supernatant Liquid from Harvested Sea Salt

Nigari (the target supernatant liquid in this study) is the liquid that is left after salt has been precipitated from seawater. In the old days, especially in the area of making salt, Nigari was used for healing scrapes and cuts. Nigari contains lots of minerals especially magnesium chloride, and more than 50 types of minerals such as calcium, potassium chloride. The magnesium helps to heal wounds.

Nigari liquid will be extracted/ collected from different solar salt production methods such as salt beds, HDPE liner and cooking facility which will be assigned as the treatments of this study (Table 3). Once the seawater evaporates to about 10% of its original volume, salt crystals begin to form (natural sea salt). Harvested sea salt from the production facilities of DMMMSU-NLUC-FRTI in Balaoan, La Union will be sun dried for 1-2 days. The liquid that flows down the bamboo basket will be collected in food grade basins, filtered in 5 microns bag or filter paper and stored for complete chemical and nutrient analysis. The liquid is expected to contain a large amount of magnesium chloride which is Nigari and has a distinct bitter taste. Samples collected will be brought to DOST-ITDI or FNRI for analysis to determine the other nutrients/chemical present in the liquid.

Table 3. Treatments for Supernatant liquid Production and characterization							
Treatment	Chemical/Nutrient Analysis	Sensory	Physical	Filth and			
	(NaCl, Protein,	Evaluation	Evaluation	Heavy metal			
	Magnesium, Calcium and		(Viscosity,	analysis			
	other trace minerals)		transparency				
			, color and				
			pH)				
Salt beds							
HDPE liner							
Cooking Facility							

#### Table 3. Treatments for Supernatant liquid Production and characterization

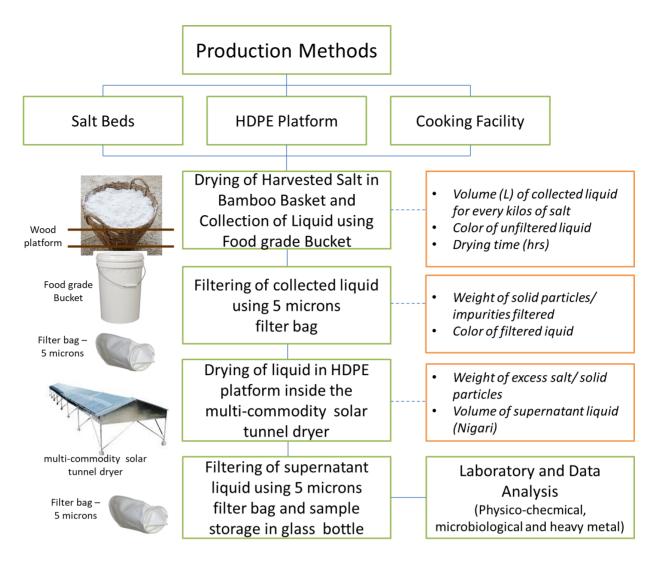
#### B. Acceptability of Nigari Liquid using Sensory Evaluation

#### **B.1** Descriptive Attribute Rating Test

After formulation, samples will be subjected to Descriptive Attribute Rating Test to determine the appropriate attribute using the work of Amerine et.al 1965 and their level of intensities. This test will be done using a structured scale from the work of Meilgaard et al 1991) with a rating of 0=None, 1= Threshold, 2= Very Slight, 3 = Slight, 4= Slight -Moderate 5= moderate, 6= moderate-strong and 7= strong. In conducting the test, a total of 10 panelists will be needed during evaluation; each panelist will be provided with coded samples at least 28 grams per treatment (Sales, 2019). Samples will be presented to the panelists and they will be provided with spitting cups and distilled water.

#### B.2 Acceptability Test

After descriptive attributes test, products will be subjected to acceptability test. A total of 100 untrained panelists will be selected during the study. During testing, consumers will be provided with 56 g of samples for three trials using metallic plate which will be coded and presented one at a time. Coding and presentation of samples in different order will be done using table of random numbers to guard against any bias (Steel, 1960). Likewise, consumers will be provided with score sheet utilizing Hedonic Scale (Jones et.al, 1955) with the rating of 9= like extremely, 8= like very much, 7=like moderately, 6= like slightly, 5= neither like nor dislike, 4 = dislike slightly, 3= dislike moderately, 2= dislike very much and 1= dislike extremely. Products will be evaluated three times based on the attributes identified during the descriptive evaluation.



Flow Diagram on the Production of Supernatant Liquid

## D. Fourier-Transform Infrared (FTIR) Spectroscopy

Salt samples will be sent to DOST-ADMATEL for FTIR analysis. FTIR will be conducted to identify and verify the chemical bonds/composition of Nigari liquid. To further standardize and characterize the Nigari liquid produced, chemical, filth and heavy metal analyses will be tested. Nigari liquid samples will be sent to DOST-ITDI to test the NaCl content, Iodine, Ca, Mg, K, Cu, , detect and count light solid impurities, and heavy metals (As, Hg, Pb, Cd).

## E. Statistical Analysis

All the collected data will be subjected to statistical analysis with ANOVA, to identify which treatments obtain significant results at the different test parameters. Any difference between the treatments means will be performed by Duncan Multiple Test using SPSS version 23. Multiple correspondence analyses using SPSS version 23 will be also used to determine/ identify which product attributes are most important to consumers and how these attributes are related to each other.

#### Study 3: Development of Salt Glazing Process for Clay Products

#### A. Design of the Salt Glazing Chamber

A gas furnace will be specifically designed and constructed for salt glazing with a chamber size of at least 0.5  $m^3$  that can provide a minimum chamber temperature of 1300°C. Refractory materials will be used as a liner so that the chamber can withstand the high temperature and corrosive condition of the process.

Sea salt produced from different production methods at the DMMMSU-NLUC-FRTI, Paraoir, Balaoan, La Union will be used for salt glazing. The sea salt will be used at different rate/amount and methods to obtain a controlled and distinct glaze on clay products. Factorial design and response surface methods (RSM) will be used to optimize the process of salt glazing and determine the condition for maximum fracture strength.

#### B. Salt Glazing Process

Test bars and clay bodies will be prepared using raw materials from Ilocos Norte at the Center for Innovative Materials in Emerging Applications (CIMEA) of MMSU. The samples will be air dried for at least 24 hours prior to the firing process. Samples will be loaded into the furnace maintaining an inch gap among samples. Pyrometric cones will be included in the chamber to ensure that the minimum chamber temperature is achieved in the process. Cone 06 (~995°C), Cone 02 (1101°C) and Cone 6 (1220°C) will be used. Upon reaching the target temperature, salt will be introduced into the chamber through a hole. Salt glazing process will be allowed for two hours. Chemical composition of the salt to the used will first be determined using AAS. The table below presents the treatments that will be used in the process.

FACTORS	TREATMENTS				
TEMPERATURE	1000°C	1100°C	1200°C		
AMOUNT OF SALT	2 kg	4 kg	6 kg		
SOURCE OF SALT	HDPE Liner	Salt Bed	Cooking Facility		

The effect of the different factors on the fracture strength of the clay body will be determined using a Universal Testing Machine available at CIMEA. The formation of the salt glaze on the surface will be confirmed from by x-ray diffraction and scanning electron microscopy (SEM) available at MMSU. The thickness of the glaze material and surface morphology will also be determined from the SEM micrograph. Resulting color of the glazed ceramic will be determined from a color chart.

## D. Statistical Analysis

All the collected data will be subjected to statistical analysis with ANOVA and any difference between the treatments means will be performed by Duncan Multiple Test using SPSS version 23.

1 <sup>st</sup> QUARTER AC	COMPLISHMENT
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Target Accomplishment/s	Actual Accomplishments	Assigned Weight	% Completion (cumulative)	%Completion (weighted)	%Completio n (Year 1)
Objective 1: Develop, standardize and characterize gourmet/flavored salts		<b>35.00</b> Y1: 25.50 Y2: 9.50			
Activity 1: Produce sea salt from different production methods (salt beds, hdpe liner and cooking facility)	<ul> <li>Produced sea salt from HDPE liner, cooking and traditional salt beds</li> </ul>	5.00	100	5	5
Activity 2: Cleaning washing and drying of raw sea salt harvested from salt production area	<ul> <li>Harvested salt were washed and sundried in modified greenhouse</li> <li>Cleaned salt were stored in temporary salt warehouse</li> </ul>	5.00	100	5	5
Activity 3: Formulation and standardization of gourmet salt from selected fish commodity (milkfish and tilapia)	<ul> <li>Waiting for procurement of multi- commodity dryer, pulverizer and ingredients for the formulation</li> <li>Target for this objective is 2<sup>nd</sup> quarter (as per workplan)</li> </ul>	15.00	0	0	0
Activity 4: Preparation and submission of samples for sensory evaluation, physical, chemical and microbial analysis, and shelf life testing	• Target for this objective is 3 <sup>rd</sup> quarter and year 2(as per workplan)	10.00	0	0	0
<b>Objective 2</b> : Develop pro byproduct (Nigari Liquid)	duct from sea salt	<b>30.00</b> Y1: 22.50 Y2: 7.50			
Activity 1: Produce sea salt from different production methods (salt beds, hdpe liner and cooking facility)	Produced sea salt from HDPE liner, cooking and traditional salt beds	10	100	10	10
Activity 2: Separation and filtration of Nigari liquid from bamboo basket during drying	<ul> <li>Collected liquid from harvested sea salt</li> <li>Temporary stored in food grade IBC tank (1 liters capacity)</li> </ul>	10	50	5	5
Activity 3: Preparation of samples for physical, chemical ( <i>NaCl, Mg,</i> <i>Mn, lodine, sulfate, Ca</i> ), and microbial ( <i>Coliform,</i> <i>Salmonella, E. coli</i> etc)	Target for this objective is 2nd quarter and year 2 (as per workplan)	10	0	0	0

<b>Objective 3</b> : Develop Salt Glazing using Sea Salt for Clay Products		<b>30.00</b> Y1: 17.50 Y2: 12.50			
Activity 1: Produce sea salt from different production methods (salt beds, hdpe liner and cooking facility)	Produced sea salt from HDPE liner, cooking and traditional salt beds reserved for salt glazing of clay products	10	100	10	10
Activity 2: Formulation and standardization of salt glazing for clay products	<ul> <li>Visited MMSU- College of Engineering clay/pottery - pot furnace</li> <li>Discussed the procedures on salt glazing</li> <li>Delivered harvested salt for trials</li> </ul>	10	20	2	2
Activity 3: Physical and chemical analysis	<ul> <li>Target for this objective is 4<sup>th</sup> quarter and year 2 (as per workplan)</li> </ul>	10	0	0	0
<b>Objective 4</b> : Report writing and publication		<b>5.00</b> Y1: 0.00 Y2: 5.00			
<b>Activity 1:</b> Consolidate data, analyze and publish	Target for this objective in year 2 (as per workplan)	0	0	0	0
					37%

Production of sea salt from different production methods was conducted prior to the development of flaked salt and fish flavored salt. Production methods include the use of traditional salt beds, HDPE liner, and cooking facility. Harvested sea salt were cleaned, packed and stored in temporary warehouse. As part of the trial, the team used the existing facility and resources available in the research facility of DMMMSU while the procurement of requested supplies, materials and equipment is ongoing.



Harvesting of sea salt produced in HDPE platform



Harvested Sea Salt and Storage

Excess liquid from harvested sea salt from different production methods were collected, stored in IBC tanks, and filtered using 5 microns bag. This process is part of the trial and standardization in the utilization of byproducts, the nigari liquid. The filtered liquids have bitter taste and texture same as the cooking oil. The next phase of the study is in the laboratory where the filtered liquid will be refined using filter paper and heating at different temperature. The team from PSU-Food Innovation Center (FIC) will visit the DMMMSU research area on March 7-8, 2024 for further discussion on the standardization of the liquid collected from the harvested salt.



Trial on collection and filtration of magnesium/potassium chloride (Nigari liquid)

The research team from DMMMSU visited MMSU-College of engineering on February 27, 2024 for the development of salt glazing for clay products. The study on salt glazing is headed by Ms. Arlene Mia Ruguian. The team inspected the existing pot furnace in MMSU and had some discussions on how to improve and execute the process of glazing.



Visitation to MMSU- College of Engineering in Batac, llocos Norte for the study on the development of salt glazing for clay products

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