BULK FISHMEAL STABILITY TRIAL: PROVIDING DATA TO AMEND THE IMSBC CODE FOR BULK SHIPPING

01 August 2018
Length of the project: 12 months

SUMMARY

IFFO has successfully provided information that has helped to amend specific clauses in the IMO's IMDG code, relevant to packaged goods. This provided information for the stabilization of fishmeal using tocopherols (250 ppm of residual levels at the time of consignment) and reduced levels of ethoxyquin (50 ppm instead of 100 ppm) at the of consignment, may be used on a voluntary basis from the 1st January 2019, and comes into the IMDG Code from 1st January 2020. However, the IMO has two shipping codes depending on the volume and the way the cargo is shipped:

- **IMDG**: International Maritime Dangerous Goods (IMDG) code adopted by the Maritime Safety Committee. Dangerous Goods mean the substances, materials and articles covered by the IMDG Code. Packaged form means the form of containment specified in the IMDG Code.
- **IMSBC**: International Maritime Solid Bulk Cargoes (IMSBC) code adopted by the Maritime Safety Committee. Solid bulk cargo means any cargo, other than liquid or gas, consisting of a combination of particles, granules or any larger pieces of material generally uniform in

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composition, which is loaded directly into the cargo spaces of a ship without any intermediate form of containment.

Commencing April 2018 and running for 12 months, IFFO, Diamante Pesquera and TASA (the companies engaged in the project via the Sociedad Nacional de Pesquera, SNP) in Peru are continuing the work that had supplied information for the IMDG Code amendment, this time for the IMSBC Code, evaluating the use of natural antioxidant Tocopherol (with Rosemary extract) and lower levels of the synthetic antioxidant Ethoxyquin.

INTRODUCTION

Varieties of animal by-products have been used as protein sources for livestock. Fish meal carries large quantities of energy per unit weight and is a source of high-quality protein and highly digestible essential amino and fatty acids. Numerous studies have shown that fish meal generally improves the growth and performance of starter and grower pigs. Fish meal and fish oils (FO) are major dietary sources of n-3 long chain poly unsaturated fatty acids (PUFA), docosahexaenoic acid (22:6n-3) and eicosapentaenoic acid (20:5n-3) (Cho and Kim, 2010). During storage of animal feed many different processes may occur which alter their initial natural properties.

First of all, lipids undergo peroxidation, the process during which they are deteriorated in a free radical autocatalytic oxidation chain reaction with atmospheric oxygen. Lipid autoxidation is a cascade phenomenon ensuring continuous delivery of free radicals, which initiate continuous peroxidation. This process results in food rancidity which manifests itself as the change of taste, scent, and color as well as decrease in shelf life of the product.

Natural or synthetic antioxidants are usually used to slow down or stop lipid peroxidation and in consequence to preserve freshness of the product (Blaszczyk et al, 2013). Ethoxyquin is a synthetic antioxidant that is included in some animal and human foods as a preservative to protect fats and fat-soluble vitamins from oxidative degradation (Case et al, 2011). However, certain food additives may cause problems. Ethoxyquin is an antioxidant about which some concerns have been raised (Bartges et al, 2008), and is in fact in a process review for reauthorisation as a feed additive according to the requirements of European Parliament and Council Regulation (EC) No 1831/2003, that sets out new rules for the authorisation, supervision and labelling of feed additives.

The lack of available antioxidants in the market whether synthetic or natural, is a clear vulnerability for the industry, and IFFO is looking at other solutions for the industry that can be used to stabilize fishmeal. Vitamin E is one of the main antioxidants used in feeds and it is fat soluble. It is added in the form of tocopherol acetate (Sampels, 2013). Vitamin E is the generic name for α-tocopherol and closely related compounds that occur in nature. There are different forms of tocopherols (α, β, γ and δ-tocopherol), but α-tocopherol is the most biologically active form of vitamin E (Chauhan et al, 2014). It is in fact α-tocopherol, the natural antioxidant under study in this IFFO work.

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OBJECTIVES

- To prove that fishmeal stored in bulk is safe when stabilized with lower (50ppm) levels of Ethoxyquin as well as providing the basis for the use of tocopherols in bulk shipments of fishmeal
- To provide data that demonstrate that fishmeal stored in bulk stabilized the same way as in IMDG code is not a MHB (Materials hazardous only in bulk) and thus the IMSBC Code can be harmonized with the IMDG code.

APPROACH

A standard Peruvian anchovy (i.e. representing a reactive fishmeal with high concentrations of fat, and polyunsaturated fatty acids) fishmeal with the same specifications as when it is sold and shipped, will be stabilised with antioxidant at variable concentrations and stored in ambient conditions in Peru. Treated fishmeal will be stored in 1-tonne bags and 50kg sacks for a period of 12 months duration as representative of shipping and storage timescales.

MATERIALS AND METHODS

Thirteen different treatments will be adopted in the trial in order to assess antioxidant efficacy at different concentrations. These fishmeals are based on one individual production line of fishmeal at Diamante Pesquera, and one at TASA fishmeal plants in Callao, Peru. For one tonne bags the treatments are as follows: Ethoxyquin treatments at 150ppm, 200ppm and 300ppm **active ingredient**; tocopherol and rosemary extract treatments at 400ppm, 600ppm, 800ppm **active ingredient (**Table 1).

Table 1: Summary of the Antioxidant Treatments in 1-tonne bags and 50kg sacks:

Treatment	Volume Sample	Concentration ppm (mg/kg)	Antioxidant	Fishery
T1	1 tonne	202	Ethoxyquin	Diamante*
T2	1 tonne	314	Ethoxyquin	Diamante*
T3	1 tonne	150	Ethoxyquin	TASA*
T4	1 tonne	300	Ethoxyquin	TASA*
T5	1 tonne	400	Tocopherols with Rosemary extract	TASA*
T6	1 tonne	600	Tocopherols with Rosemary extract	TASA*
T7	1 tonne	800	Tocopherols with Rosemary extract	TASA*
T8	1 tonne	995	Tocopherols with Rosemary extract	Diamante*
T9	1 tonne	1386	Tocopherols with Rosemary extract	Diamante*
T10	1 tonne	1935	Tocopherols with Rosemary extract	Diamante*
T11	50 kg	995	Tocopherols with Rosemary extract	Diamante*
T12	50 kg	1386	Tocopherols with Rosemary extract	Diamante*
T13	50 kg	1935	Tocopherols with Rosemary extract	Diamante*

*Fishmeal will be from the same batch
Fishmeal treatments in 1-tonne bags were treated in duplicate, i.e. two tonnes of fishmeal for each concentration. In total, there will be 20 1-tonne bags.
Fishmeal treatments in 50 kg sacks were treated with 10 replicates (sacks) per treatment. In total, there will be 30 sacks.

EXPERIMENTAL PROCEDURE

Fishmeal characteristics. The fishmeal used in this study was manufactured under standard conditions, and is representative of Peruvian anchovy fishmeals. The test fishmeal was used with the addition of the concentrated liquid product returned to the press cake, thereby including some of the soluble materials extracted during manufacture and again conforming to standard production processes.
Storage. The treated fishmeal in the 50 kilo bags was stored under normal conditions, mimicking the "ruma" storage of regular production and supply. [Ruma is the bulk storage of packaged fishmeal on site, which occurs outside in Peru due to prevailing ambient conditions of low rainfall, humidity and favourable temperatures.] The fishmeal under trial was stored at the centre of the ruma to maximise the temperature challenge to the test material. The 1-tonne bags were also stored under normal conditions.
Temperature Measurements: During the first 15 days of production, the measurement of temperature in all treatments was/will be done daily at 16:00 (4:00PM). This was/will be done in all the 20 1-tonne bags and 30 sacks used in the experiments. A daily register of the environmental temperature was/will also be recorded, establishing the difference between the internal packaged fishmeal and environmental temperature conditions. If there is a constant increase of the temperatures in any of the bags (1 ton or 50 kg) that is more than 55ºC during three days, the experiment will be stopped and will be started again with a new concentration of the antioxidant in question.
Sampling. The 50 kg fishmeal bags will have pooled samples taken from each treatment, and the 1-tonne bags sampled individually three times (top, half & bottom) will be pooled too (See figure 1).

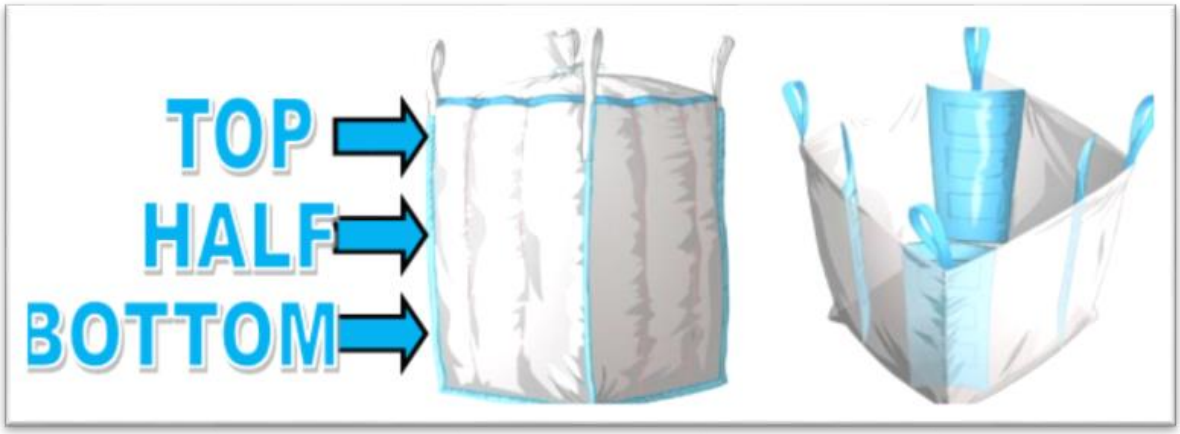

Figure 1. Depicting the Sampling of the 1-tonne sacks
The laboratory analyses for the individual samples is as described in Table 2 and the frequency of the sampling programme is as described in Table 3:

Table 2. Summary of Laboratory Analyses

ANALYSIS	METHOD	COMMENT
Remnant antioxidant concentration (A/O)	SGS-INO-ME-44:2014;REV00. Fishmeal:Total tocopheroles via HPLC; SGS-INO-ME-06 Rev.07, 2016 Fishmeal: Ethoxyquin determination via HPLC (Validated).	Measurement of the residual activity of the antioxidant during storage
Peroxide Value (PV)	SGS-PO-ME-81: 2016, Rev.05 Fish meal: Peroxide Index Determination (Validated)	PV detects the primary fat oxidation by the presence of peroxides
Anisidine Value (AV)	AOCS Cd 18-90: 2017; 7th Ed. p-anisidine value	AV detects secondary fat oxidation by the presence of secondary products
Dienes & Trienes value (DT)	AOCS Cd 7-58: 2017; 7th Ed. Polyunsaturated Acids,Ultraviolet Spectrophotometric Method.	Establishes the level of secondary fat oxidation from the peroxides breakdown
Free Fatty Acids (FFA)	SGS-PO-ME-43 Rev.06, 2016 Fish meal: Free fatty acid determination (Cold extraction) (Validated).	Measurement of the free fatty acids by enzymatic method
Ω-3 Fatty acids profile (Ω-3)	AOCS Ce 1b-89: 2017; 7 th Edition.- Fatty Acid Composition of Marine Oils by GLC.	It calculates the level of oxidation of PUFAs (EPA & DHA)
Fishmeal Temperature (°C-H)	N/A	Measurement of the fishmeal temperature within packaging.
Environmental Temperature (°C-A)	N/A	Measurement of the ambient temperature
Self-heating Test (SHT)	United Nations Manual of Tests and Criteria, Part III, 33.3.1.6 Test N4: Test Method for Self-heating substances-Testing to IMSBC Code MSC 92/26/Add.1.	Evaluates the resistance of the fishmeal to self-combustion due to oxidation at 140°C, and is a test required by both the IMDG and IMSBC Codes
Iron (Fe)	SGS-PO-ME-102: Rev.02, 2016 Metal determination in organic samples via Inductively coupled plasma optical emission spectroscopy (ICP-OES) - Microwave Oven Digestion Method. (Validated)	Evaluates the amount of iron in fishmeal (metals such as iron can act as pro-oxidants)
Biogenic amines	SGS-INO-ME-07 Rev.09, 2016 Hydrobiological products: Histamine determination via HPLC (Validated).	It reports the amount of histamine, the ingestion of higher levels of biogenic amines may result in toxicological symptom
TVN (total volatile nitrogen)	NTP 201.032: 1982 (Revised 2015) Meat and derivatives. Determination of the	Total volatile nitrogen (TVN) consisting mainly of

	ammoniacal nitrogen content (Validated 2016).	trimethylamine (TMA) and ammonia (NH3), is used as a quality criterion for fish meal and fish meal raw material (Haaland and Njaa, 1989). TVN or Total Volatile Nitrogen is a parameter that measures the presence of volatile nitrogenated basis in the raw material, or in fishmeal.
PCA (proximate composition analysis)	SGS-INO-ME-14 Rev.03, 2016 Fishmeal: Moisture determination Near-infrared spectroscopy NIRS (Validated); AOAC 990.03, 20th Ed. 2016 Protein (Crude) in animal feed; SGS-INO-ME-14 Rev.03, 2016 Fishmeal: Fat determination Near-infrared spectroscopy NIRS (Validated); AOAC 942.05, 20th Ed. 2016 Ash of animal feed (Validated); SGS-INO-ME-11 Rev.02, 2016 Fishmeal: Salt (Chloride as Sodium Chloride)-Volumetric method (Validated); NTP 204.024: 1982(Checked in 2010) Fishmeal. Sand determination.	This includes moisture content, proteins, fat, ashes, salt and sand.
Oxidation stability	Oxygen bomb	Measurement of the oxidation potential of the fishmeal sample, and hence the level of protection provided by the antioxidant

Table 3: Summary of the Sampling Programme

TREATMENT	**0 day***	**7th day,**	**15th day**	**1º month**	**3º month**	**6º month**	**9º month**	**12º month**
T1, T2, T3, T4, T5	A/O, PV,AV, DT,FFA, Ω-3, °C-H, °C-A, Fe, Biogenic amines, TVN, PCA	A/O, C-H, °C-A			A/O, °C-H, °C-A	A/O, °C-H, °C-A, SHT,	A/O, °C-H, °C-A	A/O, PV, AV, DT FFA, Ω-3, °C-H, °C-A, Biogenic amines, TVN, PCA, SHT

RESULTS

IFFO will write a final report for IMO committee with the purpose to modify the IMSBC code and harmonize it with the IMDG code. The report will also be sent to IFFO members and will be published in IFFO website and accessed through membership. We also anticipate presenting the results of this trial at both the coincident Members Meeting and Annual Conference.

BENEFITS

Members will be able to ship fishmeal at an international level using the same treatment whether in sacks/bags, containers or in bulk. This information will help to prove that lower levels of antioxidants can be used when fishmeal is shipped in bulk. Furthermore, the introduction and use of tocopherols will be promoted. This is one of several pieces of work being undertaken by IFFO on antioxidants and the aim is to provide a useful body of information to the membership that supports their business interests.

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