Rheological properties as indicator for physico-chemical processes affecting technical quality of extruded fish feed

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ABSTRACT. This paper proposes important rheological properties of fish feed pellets for withstanding mechanical stress during handling and transport. Different fish feed types are subjected to mechanical stress in the DORIS tester, to measure their mechanical durability. The same feeds are also subjected to texture analyses (texture profile analyses and stress relaxation tests), where mechanical attributes of the pellets are investigated for different orientation of the pellets. A screening test for the different mechanical properties showed that a linear model including 'cohesiveness', elastic modulus and hardness are able to describe 68 % of the variation in mechanical durability. Individual correlations against mechanical durability showed that the cohesiveness was particular important, which is a measure of the pellet's ability to return its original state after deformation. The elasticity of a pellet was also found to be of importance. Higher cohesiveness and modulus of elasticity is shown to increase the mechanical durability, while the elasticity should be low. From these results it is proposed that the pellets should be able to relax the force applied at the surface as deformation in non-elastic type behaviour. Also, a durable feed pellet should have a high elastic modulus and a viscoelastic nature promoting stress relaxation and reversible deformation.

KEYWORDS: Texture analysis, fish feed, durability, technical quality, rheology

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1 Introduction

Lack of knowledge and awareness on the drying process in fish feed production presents high energy bills to the producers, cause excessive pollution to marine and fresh water environments and elevates carbon footprints of putting fish on the dinner table.

Extruded feed is the most typical form of animal feed produced in the western world. Fish feed alone contributes with a global demand of 50-75 million tons per year and have shown an annual growth rate of more than 6% in the latest decade. It is expected that the industry will continue to grow in the coming years (FAO 2009).

Reduced technical quality of fish feed raise costs of fish farming (by reducing uptake ratios) and pollutes marine and fresh water environments. This may be the case if the pellet sinks at a velocity outside specifications, disperses when immersed in water or disintegrates during transport.

Hot air drying is an essential part of the process in the production of extruded fish feed pellets. The objectives are moisture removal, for transport and storage purposes, and to create a porous structure for subsequent oil absorption. It is acknowledged that the drying process is influencing the technical quality of feed pellets. The technical quality can be described through density, mechanical strength, pore structure and moisture uniformity in the pellet. Also, a uniform moisture distribution among pellets within the same batch is highly relevant for the description of technical quality (Carroll and Finnan 2012).

The importance of achieving a given product quality is often reflected in excessive energy use in the drying equipment, which already accounts for around 60% of the industry's thermal energy consumption. Therefore, the challenge of optimizing energy efficiency in the drying process without compromising technical quality necessitate the need for a clear trajectory from the final technical quality to the chosen set of governing drying parameters.

Thomas and van der Poel reviewed methods for assessing structural quality of pellets across food, feed and pharmaceutical branches and recommends dividing different methods into tests that determine 'hardness' or 'durability', respectively (Thomas and van der Poel 1996, Thomas, van Zuilichem *et al.* 1997, Thomas, van Vliet *et al.* 1998). Very little standardization and quality assurance exist on the assessment of physical or structural quality of extruded fish feed pellets (ANSI December 1991). Hardness tests generally comprise methods that assess either the static or dynamic break forces (Thomas and van der Poel 1996) whereas durability assess the amount of fines or breakage that occurs in a batch of product as a response to mechanic and/or pneumatic stresses (Thomas and van der Poel 1996, Aas, Oehme *et al.* 2011, ANSI December 1991). The type of stress applied is what separates the different durability test methods. In common they aim to simulate the amount and type of stress that a product will be subjected to in its lifetime. Hence, stress modes are primarily either pneumatic or mechanical. Furthermore, the extent and duration of stress could vary. The Pfost tumbling box device and the DORIS (Durability On a Realistic) test are examples of applying mechanical stress in the durability tests (Thomas and van der Poel 1996, Aas, Oehme *et al.* 2011), whereas a Holmen tester applies both mechanical and pneumatic stress. After applying a predetermined amount/duration of stress the sample is collected and subjected to a sieve analysis to give the break percentage (in one or more size indexes). One research group have even rebuilt entire sections of a bulk transportation system to accurately reproduce actual stress level and composition (Aas, Oehme *et al.* 2011). Sørensen and Aas have been engaged in trying to identify a connection between hardness and durability, using the different test methods (Sorensen, Nguyen *et al.* 2010, Sorensen, Morken *et al.* 2011, Aas, Terjesen *et al.* 2011, Sorensen 2012).

Textural analyzers were introduced first in the pharmaceutical industry for assessment of hardness and shear strength (Watano, Shimoda *et al.* 2002) and later introduced in the food industry for quantifying rheological and sensory attributes of particular fruits (Lewicki and Jakubczyk 2004, Mani, Tabil *et al.* 2006). Texture analyzers were quite recently applied in the fish feed industry for determining hardness of extruded fish feed pellets (Hansen and Storebakken 2007). Chong presented the use of texture profile analysis, TPA, for the assessment of rheological properties of dehydrated fruits at the International Drying Symposium 2012 in Xiamen, China (Chong, Figiel *et al.* 2012). Essentially TPA measures displacement vs. applied force which greatly elucidates rheological details of a product. No knowledge exists on how to relate the governing drying parameters to the mechanical, structural or rheological properties of the pellets that defines the final technical quality. Hence, it is expected that TPA can become a suitable tool in present work for identifying footprint information from the drying process, and to identify responsible physical and chemical processes that govern the formation of technical quality, e.g. starch gelatinization, glass transition, etc.

The methodology in the present work is to make correlations between conventional breakage analyses and structural details obtained from textural analyses. With knowledge of rheological properties that impact mechanical durability, the physical and chemical processes that are governing towards mechanical durability in the drying process could more easily be identified. Obviously, this is an essential precursor in the formulation of a model that predicts technical quality, coupled with a conservation model for moisture and energy, to develop a tool that at the same time optimizes energy efficiency and technical quality for the drying of extruded fish feed, which is the ultimate goal for the research project.

2 Materials and Methods

To elucidate the connection between mechanical durability and structural properties, the correlation should preferably be made using identical batches of fish feed subjected to different drying conditions, including both feed that have admitted claims as well as saleable feed of good quality. As it has not been possible to locate such a set of fish feed, the correlations are made using completely different types of fish feed to maximize contrasts in durability and structural attributes.

2.1 Materials

Nine different types of 6 mm extruded fish feed, split on 2 different feed producers, were used for the experiments. Feed types were sturgeon-, freshwater trout-, rainbow trout, turbot and salmon feeds – all products had been fully processed and were commercially available. The feed was kept air tight by double bagging and stored at 17-20 °C between the two sets of experiments. Generally, maximum 24 hours passed from the durability test to the textural analyses for the individual feed types.

2.2 Methods

Mechanical durability was assessed using the DORIS tester, Fig. 1. The DORIS tester applies mechanical stress onto pellets using a screw conveyor and a rotating propeller that accelerates the pellets into a wall. Ultimately, the pellets are collected in a sample collector bin.

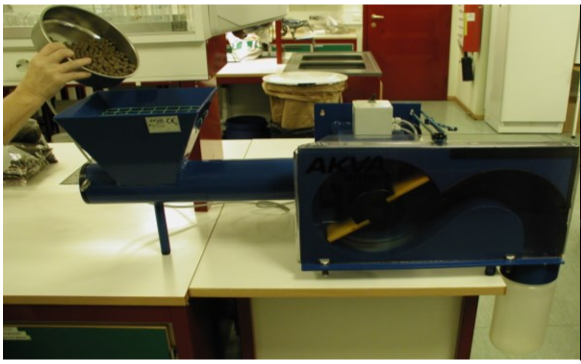

Fig. 1: The DORIS tester comprising an inlet grid, a screw conveyor, a rotating fan and a sample collector bin (EWOS Innovation 2003)

Fig. 2: The feed pellets are accelerated into a wall and collected for sifting analysis (EWOS Innovation 2003).

Approximately 200 g of sample were used for each test. The exact weight of the sample was noted before and after the DORIS test, as a small amount of the sample inevitably was lost in the test device as uncollectable dust or fines. The mechanical durability is defined as the mass fraction of sample that remains on an 8 mesh screen (2.36 mm) after 5 minutes of mechanical sifting. The weight of the sample after passing through the DORIS tester was used as the reference weight in determining the durability. The durability of each sample is determined from an average of three tests.

A Perten TVT 300-XPH texture analyser equipped with a 100 kg load cell was used to conduct textural analyses. Two different probes were used; a 50 mm cylindrical probe (Fig. 3) and a jagged kernel probe with a matching jagged female sample plate (Fig. 4). Fundamentally, a probe will move towards the sample at a pre-set speed until a predefined compression is reached, before the probe withdraws from the sample again. Force exerted against the load cell is recorded over time (resolution: 200 pps) to be used for graphical analysis. Probe type and settings will determine the type of test and the attained structural properties. The cylindrical probe was used for texture profile analysis (TPA) and for 'hold-until-time' (HUT) tests.

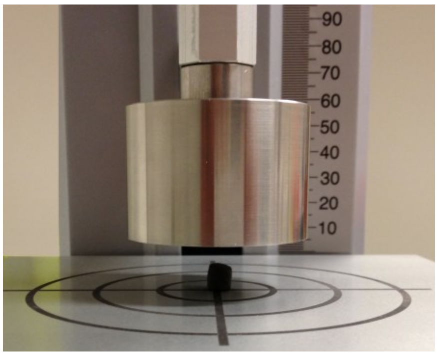

Fig. 3: Cylindrical probe

Fig. 4: Kernel probe

TPA involves the compression of a sample of at least two times in a reciprocating motion, originally developed to simulate the action of jaws. Mechanical properties typically found from TPA are cohesiveness, hardness, resilience, springiness and fracturability. These properties are well recognized by the texture analyser producers (Perten 2013) and have previously been used for determining rheological properties of dehydrated fruits (Chong, Figiel et al. 2012), cf. Fig. 5.

In a HUT test the pellet are compressed to a pre-set degree and the force registered over time will now describe the ability of the sample to endure plastic / viscoelastic deformation. A completely elastic material will retain the initial force exerted onto the load cell, whilst most extruded biomaterials will show some viscoelastic stress relaxation and therefore have elasticity less than unity, cf. Fig. 6. For both the TPA and the HUT tests it is essential that the sample (*one* pellet) does not break during the analysis.

For the HUT and TPA tests the probe was moving at 0.5 mm/s until 5% compression. For the TPA tests a 5 second pause in between cycles was used and for the HUT tests the hold time was 20 seconds. The structural attributes obtained from the HUT and TPA force-displacement graphs are averaged from 12 repetitions and were measured for pellets in both vertical and horizontal orientation.

The analyses using the kernel probe ('kernel tests') were a special case of textural analysis with the goal of determining modulus and break strength perpendicular to the radial coordinate. The probe was moving at 1 mm/s until 30% compression to achieve fracture of the 12 pellets that were analysed at a time. Modulus and break strength obtained from these tests were averaged from 5 repetitions.

The different structural attributes are computed automatically by the Perten TVT texture analyser software using the force-time curves as listed in Table 1. Note that fracturability' has been omitted in Table 1 but appears on Fig. 5. This is due to the fact that pellets do not always show this behaviour – even for the same type of pellets. Hence, fracturability is omitted in this work.

Test type	**Structural attribute**	**Calculation, cf. Fig. 5 and Fig. 6**
All	Modulus of elasticity, M	Slope of first peak (middle or last third)
TPA/HUT	Hardness, H	Maximum force
HUT	Elasticity, Q	End force / maximum force
TPA	Cohesiveness, C	Area 2 / Area 1
	Springiness, S	Time 2 / Time 1
	Resilience, R	Area 4 / Area 3
Kernel probe tests	Break strength, BS	Maximum force

Table 1: Structural properties of fish feed pellets obtained from TPA, HUT and break test with kernel probe as obtained from graphical analysis.

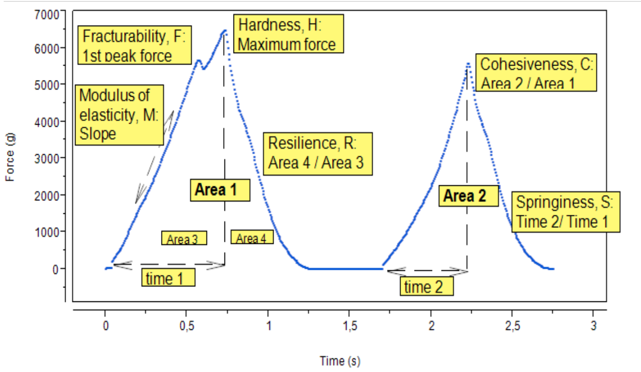

Fig. 5: Graph from texture profile analysis, TPA, with designations and calculations of structural attributes.

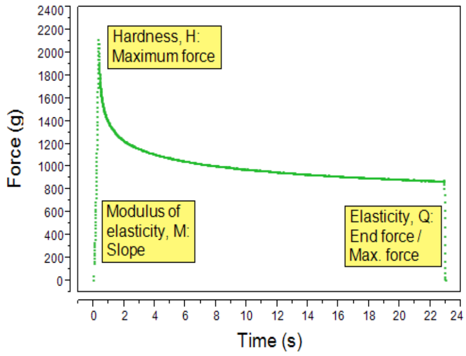

Fig. 6: Graph from 'hold-until-time' test, HUT, with designations and calculations of structural attributes.

3 Results and discussion

The structural attributes measured in each of the textural analyses were subjected to a screening test to identify those that statistically could be shown to explain some of the variation in mechanical durability. Properties that were found significant in screening tests for each of the three analyses were included in another screening test (Fig. 7). Hence, in this screening test some of the properties have already been omitted, e.g. 'springiness' was not found to describe any of the variation in mechanical durability.

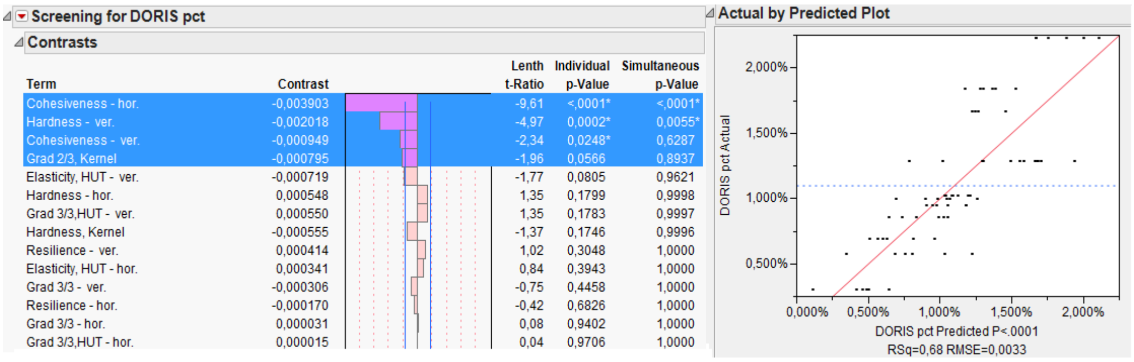

Fig. 7: Screening test for mechanical durability to identify influencing structural properties.

Interaction effects between the structural properties have been omitted in the screening test. It was found that the cohesiveness of a feed pellet explains some of the variation in mechanical durability. Cohesiveness can be interpreted as the materials ability to return to its original state shortly after a deformation. Also, the modulus of elasticity perpendicular to the radial coordinate (stress applied in horizontal position) seems significant, which is analysed in the 'kernel test' (grad 2/3, Kernel). Furthermore, the hardness (maximum force applied in vertical position in the TPA tests) also seems of some significance. A linear model fit using above mentioned structural properties are able to describe 68% of the total variation in mechanical durability.

Fig. 8 – Fig. 11 show individual linear correlations of elasticity, cohesiveness, hardness and elastic modulus against mechanical durability measured from DORIS. All correlations shown, except 'elasticity, vertical position', are found to have a slope that is significantly non-zero. In particular, the cohesiveness of the fish feed pellets explain as much as 47 % of the variation in mechanical durability. As it appears increasing

cohesiveness, elastic modulus and hardness give fewer fines in the DORIS test, and therefore higher mechanical durability. Conversely, there is an indication that an increase in elasticity will give a lower mechanical durability (increased fines content).

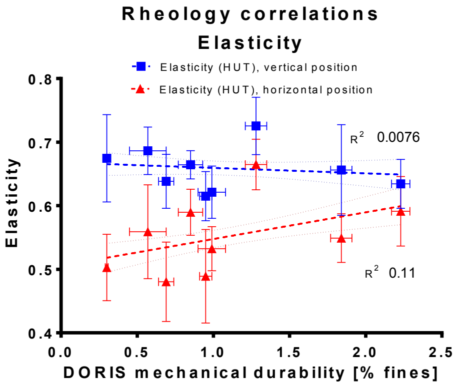

Fig. 8: Elasticity vs. mechanical durability

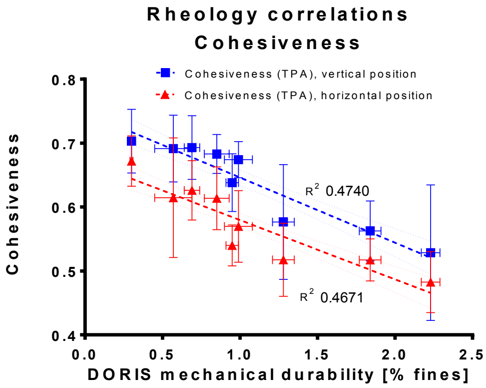

Fig. 9: Cohesiveness vs. mechanical durability

The mechanical attributes obtained from texture profile analyses are not unique material properties, but depend on the configuration of the texture analyser for each of the test methods. However, as identical test methods are applied across all samples, and by investigating Fig. 8 it is proposed that a pellet will have a high durability if the product is able to relax the force applied at the surface as deformation in non-elastic type behaviour. Importantly, as Fig. 9 shows that higher cohesiveness gives higher durability, the nature of deformation should be viscoelastic rather than viscous or plastic, i.e. the pellet should be able to return to its original state after deformation. Also, the pellet should have a high elastic modulus – results obtained in Fig. 10. support this proposition.

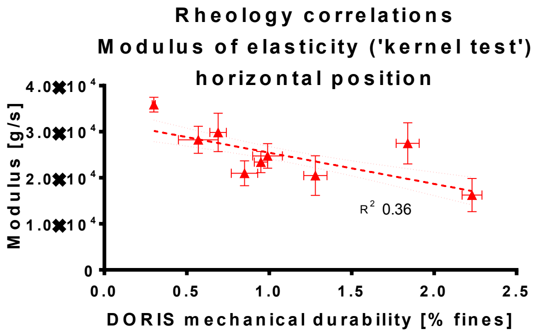

Fig. 10: Elastic modulus vs. mechanical durability

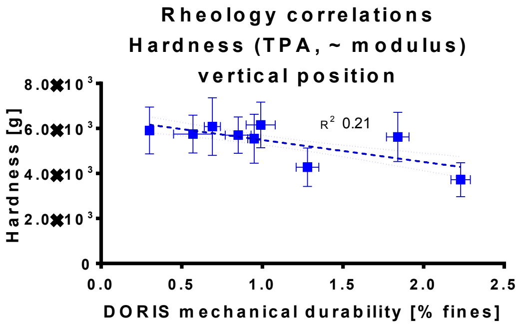

Fig. 11: Hardness vs. mechanical durability

4 Conclusions

Nine different types of fish feed was used in the correlation of mechanical durability and rheological properties. Mechanical durability was assessed using the DORIS tester and the rheological properties were determined by texture analysis as non-unique, test specific mechanical attributes. The correlations show that the cohesiveness and modulus of elasticity should be high in order to obtain good mechanical durability. Also, the pellets should be able to relax the force applied at the surface as deformation in non-elastic type behaviour. These results propose that a durable feed pellet have a high elastic modulus and a viscoelastic nature promoting stress relaxation and reversible deformation.

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