Deliverable report

Final Project Report- MICRORED

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Report

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SUMMARY
This final report is submitted as a deliverable in the MICRORED project, entitled "Reduction of Microplastic Emissions through Optimisation of Feed Pellet Conveying Systems", funded by Fiskeri og Havbruksnæringens forksningsfinansiering (FHF) under the project number 901658. The report summarises the test approached adopted in the project with relevant results. Some of the project outcomes/findings have been reported to FHF though early project deliverables.

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2) SINTEF Ocean AS
3) NORCE AS

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Sammendrag

Hovedformålet i MICRORED-prosjektet er å optimalisere transportsystemer for fôrpellets, teknologi og kostnader i oppdrettsanlegg for å minimere mikroplastutslipp og maksimere rørledningens levetid og pelletenes integritet. Prosjektet gir også anbefalinger om tiltak og beste praksis som industrien kan iverksette for å redusere utslipp av nano- og mikroplast fra fôringsrør.

En testrigg med pneumatisk transport i pilotskala ble brukt til å utføre eksperimenter som etterliknet fôrpelletstransporten i oppdrettsanleggene. Reelt fôringsrør og et kommersielt fôrpelletsprodukt ble brukt i testen. Selv om ikke testriggen kunne gjengi nøyaktig samme forhold som i oppdrettsanlegget, ble det gjort forsøk på å få resultatene så relevante som mulig for å kunne komme med nødvendige anbefalinger gjennom kontrollerte vitenskapelige eksperimentelle metoder.

Resultatene av de pneumatiske transporttestene utført i en pneumatisk transporttestrigg i pilotskala med et kommersielt fiskefôr i et standard PE-rør viser at transporthastigheten til pellets varierer i området for transportlufthastigheter fra 15 til 24 m/s. fra 1,5 til 2,2 t/t.

Dataene innhentet fra testene ble brukt til å utvikle et beregningsprogram, og rapporten beskriver potensialet ved å bruke dette til å optimalisere lufttrykkbasert fiskepelletsfôringssystemer ved oppdrettsanlegg for å minimere mikroplastdannelse. Programmet kan brukes i utvikling og optimalisering av transportsystemer for pellets. Det kan også vurderes som en del av en 'Digital Twin' om transportsystemet kobles til tilgjengelige sensorer. Den program kan kobles sammen med kontrollsystemer for å sikre best mulig drift. Programmet er basert på en oppskaleringsteknikk, og resultatene gjelder derfor kun det testede fôrpelletsproduktet, og kun for de testede rørkomponentene. Om nytt fôrprodukt og/eller nye rørkomponenter skal introduseres, bør disse testes i pilottestriggen for å oppnå relevante resultater.

Karakteriseringen av plastfragmenter ble utført av NORCE og SINTEF Ocean. Prøvene, som ble tatt fra det pneumatiske transportsystemet, påpekte dannelsen av mikronstore partikler. Observerte nivåer var avhengige av hvor prøvene ble samlet inn i transportsystemet. Prøvenes tydelige fragment-størrelsesfordelingsmønster identifiserer en mulig rolle for fettlaget som belegger den indre delen av fôringslangen under standard fiskefôrtilførsel. Basert på de observerte resultatene er det ingen plastfragmenter under 10 µm (nanometrisk størrelsesfraksjon) i de granskete prøvene. Det er imidlertid viktig å merke seg at det er begrensninger med denne tilnærmingen. Den primære begrensningen er knyttet til mangelen på referansekjemikalier til den valgte tilnærmingen. Videre kan prøveprepareringsprosedyren for både den enzymatiske oppløsningen og ekstraksjonen ha forårsaket nedbrytning og fordampningstap fra prøvematerialet.

Den morfologiske undersøkelsen av det eroderte røret indikerer at erosjonen av fôringsrøret varierer med posisjonen til røret i transportsystemet. Forskjellig posisjonerte rør utsettes for varierte sammenstøt fra fôrpellets. Ved utsiden av rørbøyningen blir flere pellets akkumulert, noe som fører til kraftig erosjon. Innholdet av fett som belegger den indre delen av fôringsslangene spiller en rolle i størrelsesfordelingen til de genererte plastfragmentene under simuleringen av transportdistribusjonen.

Project summary

The main objective of the MICRORED project is to optimise the feed pellet conveying systems, technology and costs in fish farms to minimise microplastic emissions and maximise pipeline lifetime and pellet integrity. The project also provides recommendations on measures and best practices that the industry can implement to reduce emissions of nano- and microplastics from feeding pipes.

A pilot scale pneumatic conveying test rig was used to perform experiments mimicking the feed pellet transport in the fish farms. The actual pipeline and a commercial feed pellet quality were used for the test. Though the test rig was not exactly displaying the real-life pellet transfer system of the farm, attempts were made to obtain the results as relevant as possible to make necessary recommendations through controlled scientific experimental methods.

The results of the pneumatic conveying tests carried out in a pilot-scale pneumatic conveying test rig with a commercial fish feed and a standard PE pipe show that in the range of conveying air velocities from 15 to 24 m/s the transport rate of pellets varies from 1.5 to 2.2 t/h.

The data obtained from the tests were used to develop a calculation programme, and the report describes the potential of using it to optimize air-based fish pellet feeding systems at fish farms to minimise microplastic generations. The program can be used in pellet transfer system design and optimization, and it can also be considered as a part of 'Digital Twin' of the transfer system connecting with available sensors. It can be coupled with control systems to make sure the optimized operations. The program is based on the scale-up technique, thus, the results valid only the tested quality of pellets. Only the tested pipe components can be simulated. To include new feed quality and/or new pipe component, those should be tested in the pilot test rig and acquired necessary results.

The plastic fragments characterization exercise performed by NORCE and SINTEF Ocean on samples obtained from the pneumatic convey system pointed out the formation of microns sized particles. Observed levels were dependant on where samples were collected in the conveying system. The samples distinct fragment size distribution pattern identifies a potential role of the fats layer coating the inner section of the feeding hose during standard fish feed supply. Based on the observed results there is no plastic fragments below 10 µm (nanometric size fraction) present in the observed samples. However, it is important to note that there are limitations with this approach. The primary one is linked to the lack of reference chemicals and the sensitivity study of the presented approach. Furthermore, the sample preparation procedure for both the enzymatic digest and extraction may have caused degradation and evaporative losses, respectively, of the target compounds.

The morphological investigation of the eroded pipe indicates that the erosion of the pipe is varying upon the position of the pipe in the convey system, as different positioned pipe received varies impacts from feed pellets., and at the elbow of the bend, more pellets are accumulated, leading to severe erosion. The content of fats coating the inner part of the feeding hoses play a role in the size distribution of the generated plastic fragments during the convey distribution simulation.

1) Project background

Plastic equipment used in the fisheries and aquaculture industry contributes to emissions of microplastics into the sea, which may potentially have negative consequences for the marine environment and living organisms. The release of microplastics from the fish feeding pipes has been recognized as one of the contributing factors to the pollution of the sea water, with implications for seafood safety and potentially human health, lowering the consumer confidence in seafood products.

In larger fish farms, the fish feed is typically transported by means of compressed air from the storage point to several fish cages through a network of transportation pipes that are typically made of plastic, mainly HDPE (high-density polyethylene) (Figure 1).

Figure 1: Centralized feeding system *(photo by Chandana Ratnayake).*

The use of unnecessarily high air flow volume rates accelerates the pellets to a high velocity, causing hard impacts with the internal wall of the pipe, especially in bends and curved sections, with directional change of pellet conveying path. Depending on the fish feed properties (hardness, shape, size, surface texture), this can result in potential problems with negative economic and environmental consequences:

A. excessive erosion (abrasion) of the pipe surface and faster wear that leads to more frequent replacement of the pipeline;
B. significant breakage of the pellets leading to local pollution and loss of valuable and costly feed; and
C. higher rates of microplastic release into aquaculture facilities and further into the wider environment.

On the other hand, the use of too low air velocity may lead to pipe blockages and pellet breakage due to compressive stress. Optimization of feeding system operating parameters is therefore key for ensuring minimal release of microplastics from the feeding pipes, maximising the lifetime of the pipeline and for delivering intact, undamaged pellets to the fish.

MICRORED, fully entitled "Reduction of Microplastic Emissions through Optimisation of Feed Pellet Conveying Systems", is a research project, fully funded by the Norwegian Seafood Research Fund (Fiskeri- og havbruksnæringens forskningsfinansiering – FHF). The project has been funded through the FHF 2020 call:

"Tiltak for å redusere utslipp av plast fra sjømatnæringen"

1. Delmål 3: "Fremskaffe kunnskap om tiltak og beste praksis som næringen kan iverksette for å redusere utslipp av nano- og mikroplast fra fôrslanger."

The main objective of the MICRORED project therefore to optimise the feed pellet conveying systems, technology and costs in fish farms to minimise microplastic emissions and maximise pipeline lifetime and pellet integrity. The project will also provide recommendations on measures and best practices that the industry can implement to reduce emissions of nano- and microplastics from feeding pipes. This is achieved through the following sub-objectives:

1. To evaluate the effect of air velocity and pipeline configuration (bend radius) on pipe wall erosion for selected fish feed qualities.
2. To quantify the amount of micro- and nanoplastic (MNP) fragments from objective A and characterize their physical properties (size, shape).
3. To map the erosion pattern and evaluate the evolution of erosion with application time.
4. To implement the results in a simulation software for a selected industrial site to demonstrate how the feeding system can be optimized.
5. To disseminate the learning from the project and present the methodology for optimization of the feed pellet conveying systems to the fish farming community.

The project work will be divided into 4 WPs, shown schematically in Figure 2.

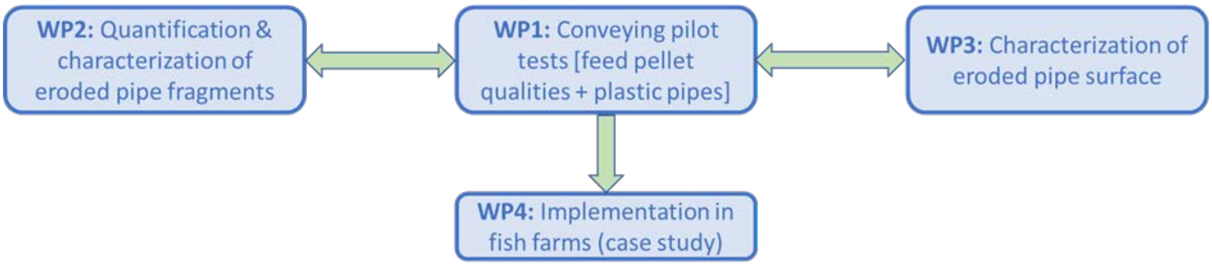


Figure 2: Schematic outlining the work packages in MICRORED and their connections

This document is the final report of the MICRORED project. It provides an overview of previous studies, investigating the release of microplastics from the feeding systems, and describes the findings and results from the experimental tests carried out under work packages 1-4 over the project period (15/01/2021 – 31/12/2022).

2) Previous work on microplastics from feeding systems

Research studies investigating the release of microplastics from the fish feeding pipes as a consequence of pipe wear are scarce. The first report on potential release of microplastics from the feeding pipes has been published by Naturvernforbundet in 2017 [1]. A rough estimate of release of microplastics from the feeding pipes was obtained by weighing five 2-m long sections of worn out, discarded feeding pipes. The pipe sections weighed from 1320 to 1490 g per meter while a new pipe typically weighs around 1900 g per meter. From these measurements, it has been concluded, that between 410 to 580 g plastic per meter is potentially lost during the pipe's lifetime (assumed 5 years), corresponding to a release of 325 tonnes of microplastics into the sea every year from Norwegian fish farms. However, these estimates were considered uncertain because they were based on measurements from only five pipe segments, without knowing the history of their use and whether the pipes can be considered a representative sample. Therefore, Naturvernforbundet informed the Ministry of Climate and Environment about their findings and requested a systematic investigation to obtain an accurate estimate of microplastic release from the feeding pipes.

To measure the amount of pipe wear and microplastic emissions in real fish farm systems is however challenging and currently, there are no established standard methods to do such measurements. The next study, carried out under the FHF funded project HavPlast [2], has taken a theoretical approach and attempted to estimate the release of microplastics from the feeding pipes based on mathematical simulations [3]. An erosion model originally developed for sand erosion in a gas pipe in the oil & gas sector was adopted and used to estimate the amount of erosion caused by pellets in a feeding pipe as a function of velocity and impact angle (pipe curvature). The pipeline geometry (length, curvature, degree of pipe twisting, etc.) have been derived from statistical analysis based on photographs taken during visits to fish farms as well as from satellite pictures (Figure 3). Operating parameters and other technical specifications, such as conveying velocities, pipe thickness, pellet size, transport rate, etc., have been obtained directly from fish farmers or producers of feeding pipes and fish feed.

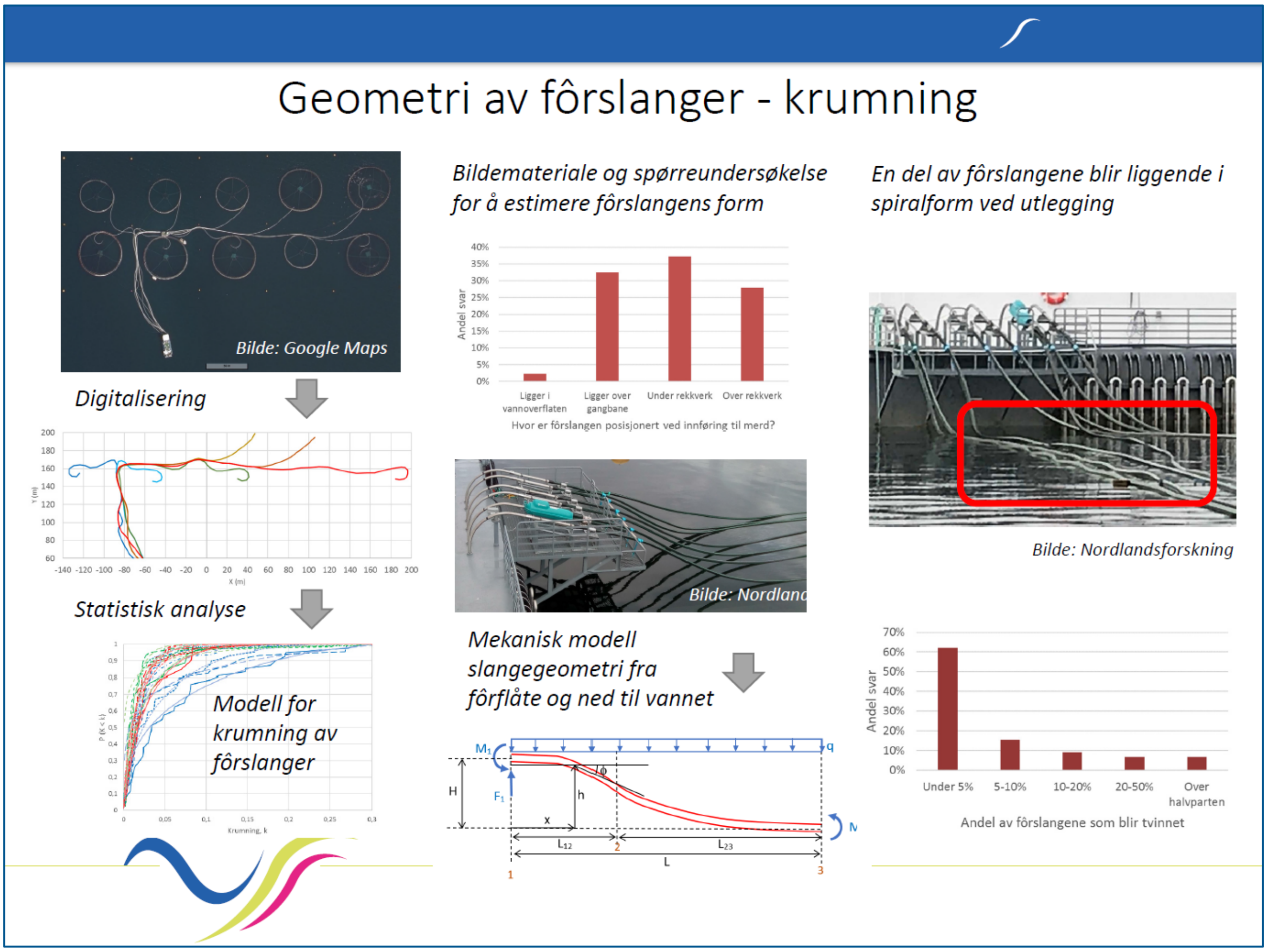


Figure 3: Illustration of how photographs were used to derive the geometrical parameters of the feeding pipes for the use on the model (source: Nordlandsforkning [4]).

Interviews with fish farmers indicated that a feeding pipe has a lifetime in the range of 18-24 months (which is considerably shorter compared with assumptions by Naturvernforbundet [1]) and overall, about 1/3 of feeding pipes develop a hole due to wear during their lifetime. The model was thus calibrated to provide the same proportion of feeding pipes that develop a hole during their lifetime, as reported by the fish farmers. The amount of erosion calculated for a single feeding pipe was then scaled up to provide a total estimate of microplastic emissions from feeding pipes from Norwegian fish farms. The simulations indicated that national emissions of microplastic from the feeding pipes range between 10 to 100 tonnes a year, with an average value at around 30 tonnes a year. However, it was concluded that there is a large uncertainty in the results due to lack of representative input data as well as no physical measurements that could be used for calibration and validation of the model. It was also pointed out that the model, originally developed for sand particles, might not be suitable for estimating erosion by fish feed pellets due to different particle properties of pellets (size, shape, hardness, etc.) with respect to sand [3], [4].

The following FHF funded project TrackPlast [5] attempted to estimate the amount of microplastic emissions from feeding pipes based on experimental tests. The tests were conducted in 5-meter-long pipe sections of an HDPE feeding pipe. Artificial pellets made of clay, mimicking the physical properties of real fish feed pellets (density, weight, dimension, abrasion, etc.), were transported through the pipes twice a day (6h + 6h) for one week under constant conditions. The report describes the clay pellets to have an uneven spheroid shape, but there is no information regarding the pellet size. The effect of age was investigated by testing new and aged pipe sections. The tests were conducted in straight sections or sections that were curved to reach a 10° angle. The test conditions were kept constant in all the tests as indicated in Figure 4. Approximately 2 tonnes per day of artificial clay pellets were transported, with 12 hours of daily operation corresponding to a transport rate of 0.167 tonnes/h. The generated microplastic particles were analyzed for particle size distribution and shape [5]. Erosion was determined as weight loss by weighing the pipe sections before and after the test.

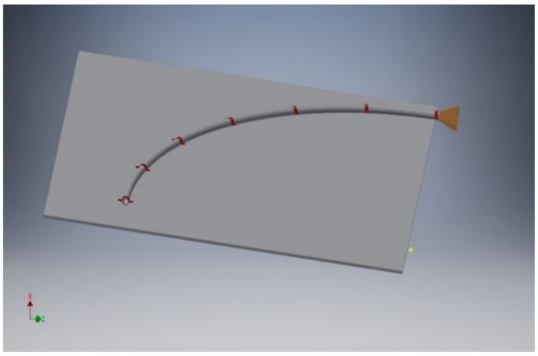
Characterizing input sources through the fish feeding line

NORCE

To simulate different naturally occurring shapes of the pipes in an aquaculture site:

- A new and an aged pipe were placed on a plane testing table and curved off by a horizontal plane to reach a 10° angle
- A new and an aged pipe were kept straight
- Pipes were weighed before and after the experiment. Pellets were pushed through the pipes twice a day (6h + 6h).

Generated debris was analyzed for particles size distribution ……



Parameter	Value
Air speed	20 m/s
Pellet speed	15 m/s
Pressure	0.5 bar
Temperature	70 °C
Blower system - Air volume	200 m^3/h
Blower system - pressure	1 bar

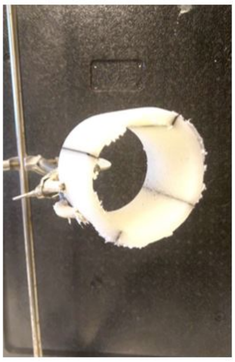


Figure 4: Abrasion tests conducted in the TrackPlast project (source: NORCE [6]).

The tests showed that after one week of operation the weight of the pipe sections decreased by 5-14 g, corresponding to an average loss of 0.1 to 0.4 g/meter/day at the given experimental conditions. The highest weight loss was measured in curved aged pipes while minimum weight loss occurred in new straight pipe sections, indicating that aging and pipe curvature increase erosion. The measured weight loss corresponds to a theoretical release of 150 to 569 kg of microplastics per year per aquaculture site, assuming a site with an average length of the feeding pipes of 4000 m. With 800 aquaculture sites in Norway [1], the national release of microplastics from the feeding pipes ranges from about 120 to 455 tonnes/year.

The MICRORED project aims to investigate erosion in the HDPE feeding pipes through experimental testing in a pilot-scale pneumatic conveying set-up and attempts to estimate the degree of erosion using commercial fish feed pellets. Since there is no established methodology for the measurement of erosion in the fish feeding pipes, this study attempts to make a step towards development of suitable methods, while evaluating the feasibility and limitations of physical tests and ways for future improvements.

The pilot-scale tests, the analysis of MNPs, the characterization of eroded pipes and recommendations related to optimization of pneumatic fish feeding systems are further described in the respective WPs in the following sections.

3) WP1- Conveying pilot tests

a. Objectives

In WP1, it was proposed to carry out pneumatic conveying tests in the pilot-scale test set-up available at SINTEF Tel-Tek using a commercial fish feed and standard HDPE pipe. The main objectives of WP1 were:

- To characterize the flow behavior of fish feed pellets during pneumatic transport under different conveying conditions and to obtain relevant data (pressure development along the line, volume flow rate and transport rate) as basis for the development of a calculation programme for optimization of pneumatic conveying systems at fish farms in WP4

- To estimate the amount of erosion generated during pneumatic transport for fish feed pellets in an HDPE pipe under selected conveying conditions and to collect samples for further analyses in WP2 (analysis of generated MNPs) and WP3 (analysis of eroded pipe surface)

These experimental tests aim to provide a realistic picture of the amount of microplastics potentially released into the environment from the feeding pipes of an industrial scale fish farm and to identify optimal conditions for pneumatic transport with minimized erosion.

b. Materials and methods

i. Conveying test rig

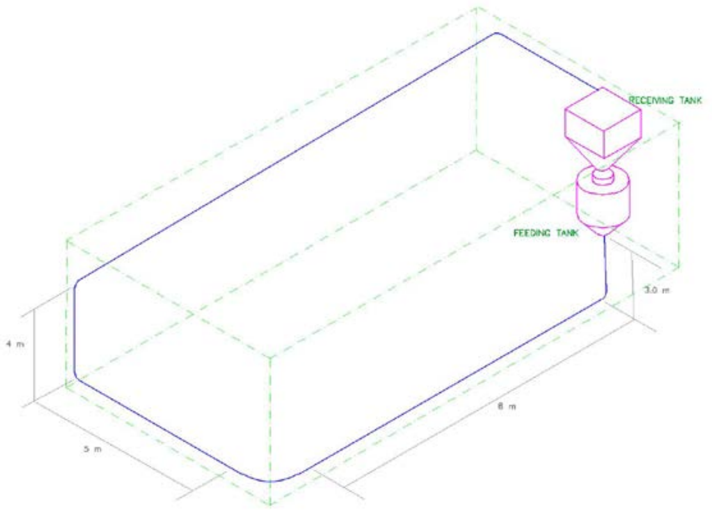
The pilot scale test rig was set-up at Powder Hall in SINTEF Tel-Tek (STT) mimicking a transport system at a fish farm. A schematic diagram of the rig configuration is shown in the Figure 5A.

The test rig consists of a discharge tank of 2.5 m^3 capacity, a receiving tank, which is mounted on a special arrangement of load cells to monitor the weight accumulation during the tests. The pipeline is a standard HDPE pipe, kindly provided by HallingPlast, with an external and internal diameter of 90 and 75 mm, respectively, and approximately 40 m in length. The feeding from discharge tank to conveying pipeline is arranged through a rotary valve. The conveying line forms a closed pneumatic transport circuit by placing the receiving tank on top of the blow tank so that the pellets under testing can be repeatedly transported without taking them out of the test rig (Figure 5B).

The air supply is received from a combination of a screw type air compressor and a drier/air cooler, the pellets are thus conveyed with compressed air that is dried to a dew point of about 4 °C. The pressure and volume flow rate of supply air is controlled by a regulator. The transport rig is equipped with facilities for continuous online data logging and visualising of data like air pressure at various locations, material transport rate, and air volume flow rate on a real time basis. The data acquisition and analysis are undertaken with the help of a software program of the LabVIEW® package. All tests have been carried out at ambient temperature.

As illustrated in Figure 5A, the pneumatic conveying rig contains four 90° bends, a vertical section and several horizontal sections of different lengths.

A



B

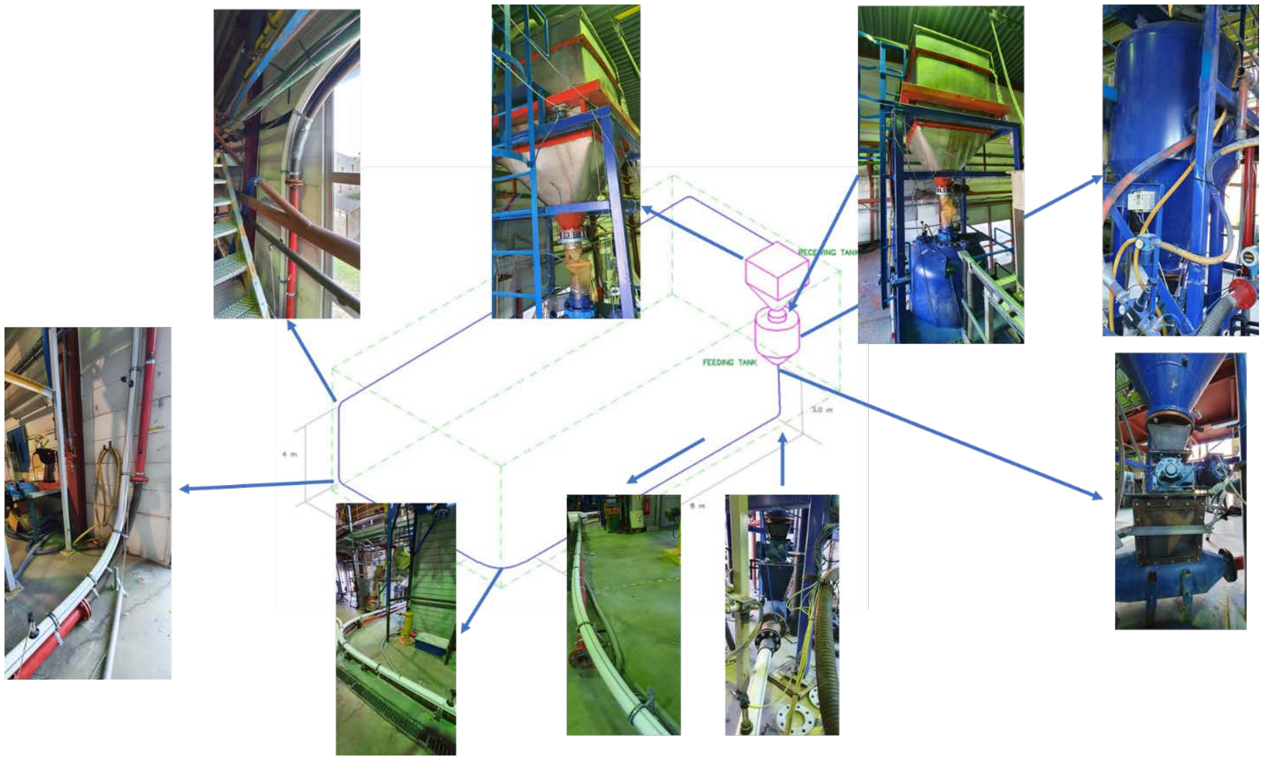


Figure 5: Schematic view of the conveying test rig (A), pictures showing different parts of the test rig and equipment (B)

ii. Feed pellet characteristics

The pellet quality named 'Protec' was purchased from Skretting. Their characteristics are given below:

Table 1: Characteristics of tested fish feed pellets

Pellet quality	Height (mm)		Diameter (mm)		Pellet Density (kg/m3)	
	max	min	max	min		
Protec	10,0	9,1	10,1	9,0	1,18	

iii. Measurement of erosion by weight difference

The degree of erosion is typically measured gravimetrically, meaning the specimen is weighed before and after exposure to erodent. In this study, erosion is studied in one of the 90° bends (Figure 6) that can be detached from the pipeline and weighed before and after the tests to determine the weight difference.

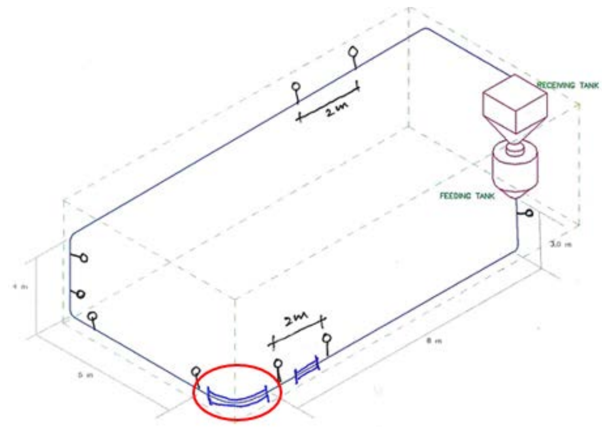


Figure 6: Erosion is studied in the 90° bend marked by the red circle.

As will be shown later, for accurate weight measurements, it is important to ensure that the pipe is clean. The pipe's inner surface can become contaminated by oil and dust from the pellets that are released during the test. To clean the pipe, a mixture of NaOH and KOH (approx. concentration 10-30 wt. %) was used. This was a commercial solution purchased in a local plumber shop for cleaning clogged pipes in households.

Before application, the purchased NaOH/KOH mix was diluted approximately 10 times. After the tests, the bend piece was disconnected from the pipe, fixed in a position (with open ends aiming upwards), filled with the diluted cleaning solution and let stand overnight. The next day the bend was rinsed with plenty of water. To remove the last remains of the pellet residues, a cleaning soft sponge (Figure 7) was pushed through the pipe. After using the ball, the pipe was rinsed with water again and let dry until next day when the weight of the bend piece was taken.

Figure 7: A cleaning sponge kindly provided by AKVA group for cleaning of feeding pipes.

iv. Measurement of pipe thickness

A hand-held device for the measurement of pipe thickness, Olympus 45MG Ultrasonic Thickness Gauge, was used to determine pipe thickness at specific locations along the bend piece where erosion was studied. The instrument has an accuracy of +/- 0.10 mm with a standard resolution of 0,01 mm.

Figure 8: Olympus 45MG Ultrasonic Thickness Gauge[1].

Pipe thickness was measured manually on clean surface before and after tests in multiple points along the bend piece. A simple sketch in Figure 9 illustrates the measurement positions. At each point, four measurements around the pipe circumference were taken (top, bottom, inner side and outer side of the bend wall).

[1] https://www.olympus-ims.com/en/45mg/

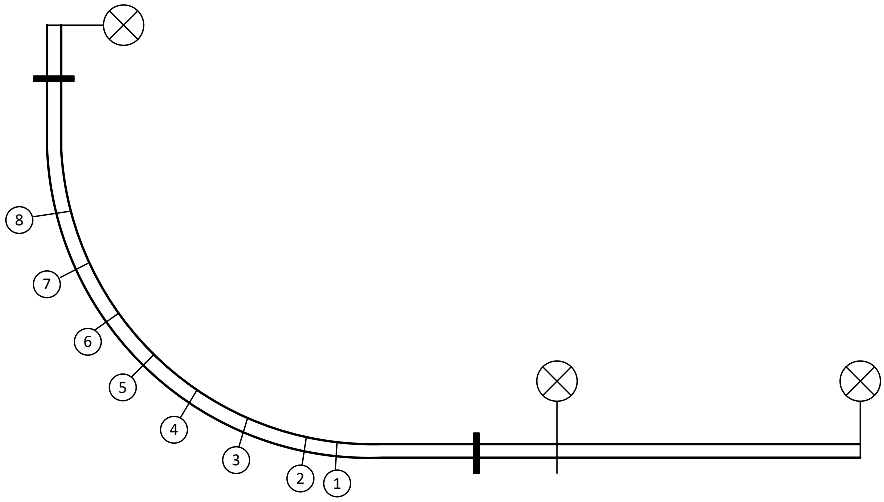


Figure 9: Pipe thickness measurement points at 8 locations along the bend. Four measurements (top, bottom, inner side and outer side) around the pipe circumference taken at each location.

v. Sample collection

Samples for analysis of MNPs (WP2)

Samples for analysis of MNPs were taken after erosion tests from three locations as illustrated in Figure 10. Erosion tests resulted in a significant pellet degradation and formation of an oily dust deposit inside the feeding pipes. The sample of the deposit was taken directly from the inside of the bend piece after detaching it from the line. The cleaning solution after it has been used for cleaning of the bend piece was also used as a sample for further analysis of MNPs. Further a sample of used pellets has been taken from the receiving tank. The fine dust collected in a container below the bag filter was also used a sample for analysis MNPs.

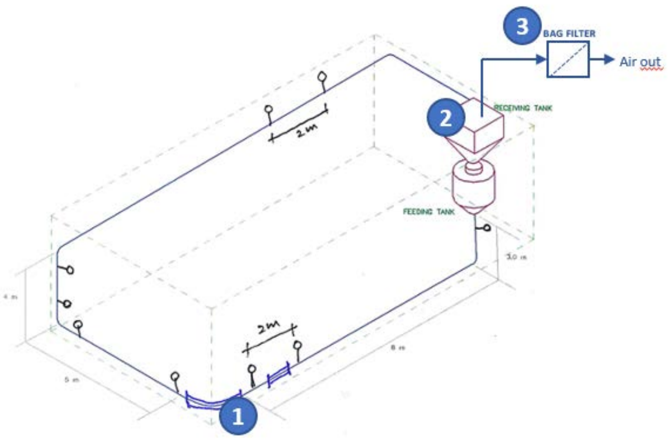


Figure 10: Illustration of sampling points for analysis of produced MNPs during erosion tests.

Samples for characterization of eroded pipe (WP3)

After erosion tests, the bend piece used in the tests was cut into small sections that were further analysed as described under WP3.

c. Results

i. Characterization of pellet flow behavior – pressure tests

To characterize the flow behavior of fish feed pellets during pneumatic transport, we have carried out a series of pressure tests. During these tests, the volume flow rate and the pellet feeding rate by the rotary feeder were varied in a systematic manner to create different conveying conditions (solids loading ratio, conveying velocity) while collecting pressure data along the line and transport rate data from the load cells installed on the receiving tests. The conveying velocity varied in the pressure tests from 15 to 24 m/s and the measured transport rate was between 1.5 to 2.2 t/h, which correspond to typical values used by fish farm operators. The methodology and the results of the tests were described in more detail as part of deliverable **D1.1 Calculation program to optimize system performance**. The data collected during the tests, especially information about pressure drop across different pipe elements such as bends, straight horizontal section and straight vertical section, will provide basis for the development of a calculation programme that can be used for optimization of full-scale pneumatic systems at fish farms.

It is important to note that the purpose of the tests is to demonstrate how a pneumatic conveying system at a fish farm can be optimized in order to avoid the use of unnecessary high velocities that can result in significant erosion and pellet degradation. The flow behavior of particles in pneumatic conveying is in general affected by the properties of the particles (particle size, size distribution, density and shape). Therefore, the data collected during the tests is only valid for the feed that was used during the experimental tests (Protec) or any feed that has the same (or very similar) properties as shown in Table 1.

The data obtained from the pressure tests have been used to make the calculation program for design and optimization of feed transfer systems in fish farms. WP4 provides more information.

ii. Pellet degradation during pressure tests

As explained in Section b.i, the pellets are fed into the line by a rotary valve from the blow tank and transported through the conveying line to the receiving tank. After transporting all the pellets from the blow tank to the receiving tank, the receiving tank outlet is opened through a pneumatic valve, the pellets are discharged through a transparent hose connection to the blow tank and a new test starts. The pellets are thus recirculated in the conveying loop several times before they are taken out of the system and replaced by fresh pellets. Pellet degradation during the pressure tests was monitored visually between the tests during the discharge of the pellets from the receiving to the blow tank. After conducting all the pressure tests to collect the necessary data from WP4, the pipeline was opened by dissecting out the bend piece that was to be monitored during the erosion study (Figure 6). The view in the bend and the neighboring straight sections shows that the pellets underwent some degradation during the pressure tests inside the 90° bend, as displayed by the oily dust deposit at the outlet of the bend and the straight section after the bend (Figure 11C and D, respectively). Despite recirculation of the pellets, the straight section before the bend remained clean, indicating low oil seepage from the pellets during the tests and therefore only minor pellet degradation (Figure 11A and B). This is also confirmed by the photographs of the fresh pellets and the used pellets after the pressure tests in Figure 12. This minor pellet degradation did not severely change the pellet properties and had no significant effect on the quality of data collected during the pressure tests, making them directly applicable to real operation of full-scale pneumatic conveying systems at fish farms.

A B C D

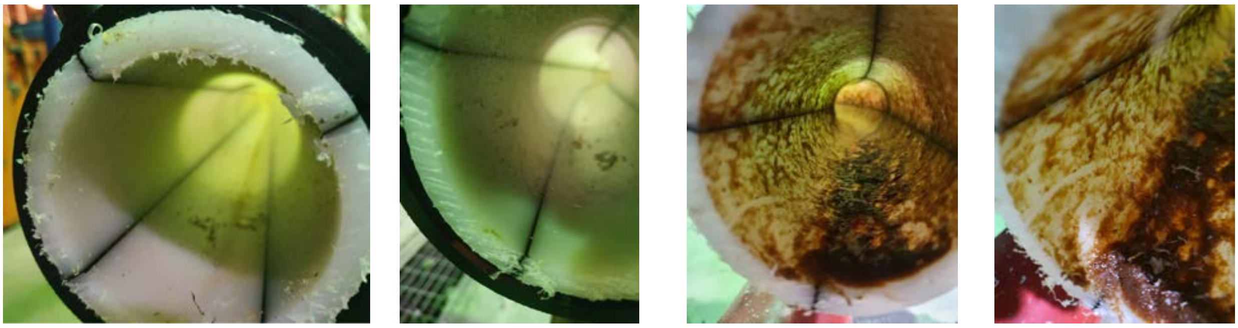


Figure 11: Photographs taken after dissecting the pipe bend for the erosion study: a view into the straight line before the bend (A), into the bend inlet (B), into the bed outlet (C) and into the straight section after the bend (D).

A B

Figure 12: Photographs of fresh (unused) pellets (A) and used pellets taken out of the pneumatic conveying test rig after finishing all pressure tests (B).

iii. Estimation of erosion during conveying tests – erosion tests

Impact erosion or wear of the pipe's inner surface is often a negative side-effect of pneumatic conveying especially when conveying abrasive materials. Erosion is typically highest in areas of directional change of flow, such as in elbows, bends, diverters, or valves. As the particles negotiate the curved section, they often collide with the outer wall of the pipeline, which can result in surface damage and material removal from the pipe's surface. With time and under continuous exposure, pipe hole may eventually occur, requiring replacement of the pipe.

Erosion is dependent on many factors including:

- Particle properties (size, shape, hardness, etc.)
- Pipe material properties (hardness, ductility, roughness, etc.)
- Pipeline geometry (curved sections and bend radii affecting the impact angle)
- Operating conditions (velocity, temperature, humidity, etc.)

Among the most critical factors is impact velocity and impact angle and therefore, design of the pneumatic conveying system and optimization of operating conditions are often key in minimizing the effect of impact erosion.

Impact erosion in pneumatic conveying is typically studied in bench-scale erosion testers. The working principle is based on exposing a test specimen (e.g., a sample of pipeline material) to a jet of air carrying erosive particles. The amount of erosion is determined by weighing the test specimen before and after test. The advantage of bench-scale erosion testers is that erosion conditions (impact velocity, impact angel, type of erosive particles, target material) can be easily varied and many tests can be performed in short time. Extrapolation of the results generated in bench-scale test rigs to real industrial systems is however not straightforward and research is ongoing to develop suitable models. In addition, the use of standard bench-scale erosion testers is limited to fine powders, such as cement, sand, etc. Currently, there is no established standard method or equipment available to measure erosion of fish feed pellets in feeding pipes during operation at fish farms.

To determine the amount of erosion in feeding pipes, it is necessary to estimate the pipe material removal. This can be done by installing pipeline segments that can be disconnected from the main pipeline after the tests to measure the resulting weight loss. Alternatively, pipe thickness can be measured at specific locations along the line by ultrasound sensors. In this study, we focused on erosion measurement in the 90° bend as indicated in Figure 6.

Fabrication of detachable bend segments

As explained in Section ii, once the pressure tests were finished, the pipeline was opened by cutting out a bend segment (Figure 11A). Flanges were mounted on both sides of the used bend segment and at both ends of the adjacent straight pipe sections so the bend piece could be re-connected with the pipeline. An additional bend segment with flanges was fabricated using a virgin pipe (Figure 13). The detachable bend segments were used in subsequent erosion tests to estimate the degree of erosion during pneumatic transport of fish feed. Pressure transducers are installed before and after the bend to monitor pressure over the bend during the tests.

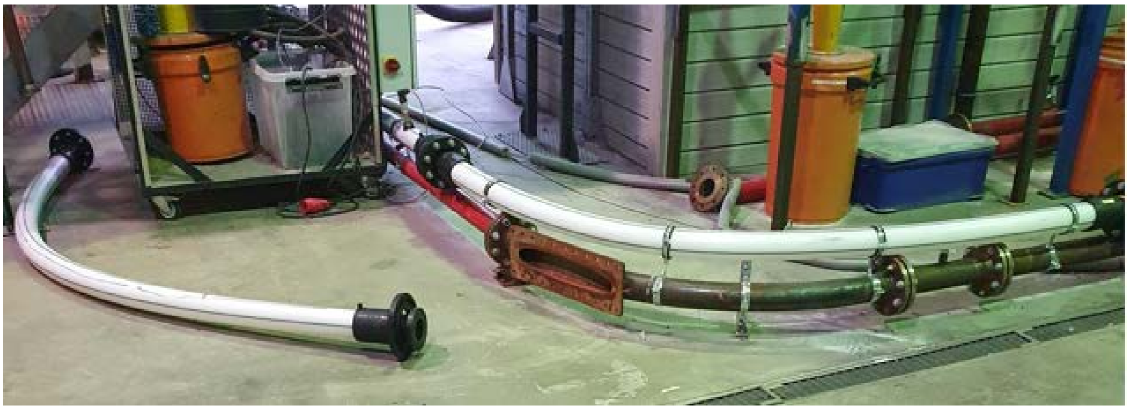


Figure 13: Two bend segments with flanges fabricated by Bilfinger.

iv. The effect of impact velocity

Based on communication with fish farmers and feeding system operators, the typical air velocities used at fish farms lie between 12 and 20 m/s. Occasionally, air velocities as high as 30 m/s may be used but this is not recommended by the providers of the feeding systems due to risk of high erosion and high pellet degradation. Based on this, 28 m/s - 32 m/s velocities were chosen for the erosion tests.

High velocity erosion tests

Prior to the tests, the clean bend segment was weighed, and the value recorded. About 180 kg of fresh feed pellets were loaded into the blow tank. During pneumatic transport, the compressed air that transport the material expands, which results in increasing actual volume flow rate and thereby, increasing actual air velocity along the pipeline. The volume flow rate during erosion tests was set to achieve an actual velocity of about 30 m/s inside the detachable bend piece. The rotary valve was set to a feeding rate of about 2 tonnes per hour, which is a typical transport rate used at fish farms. The pellets were transported through the conveying loop between the feed and the receiving bend seven times. After the first seven rounds, the used pellets were replaced with fresh pellets and the procedure repeated for another seven rounds.

The pressure along the pipeline, the volume rate flow rate, and the weight of the transported pellets into the receiving tank were monitored during all the tests. In the first and second test, the conditions were quite stable, maintaining a constant air velocity during the tests in the range of 28-32 m/s (Figure 14). The second test was interrupted after transporting the first 100 kg of pellets into the receiving tank, but the conditions were also quite stable when re-commencing the test to transport the remaining amount of the pellets.

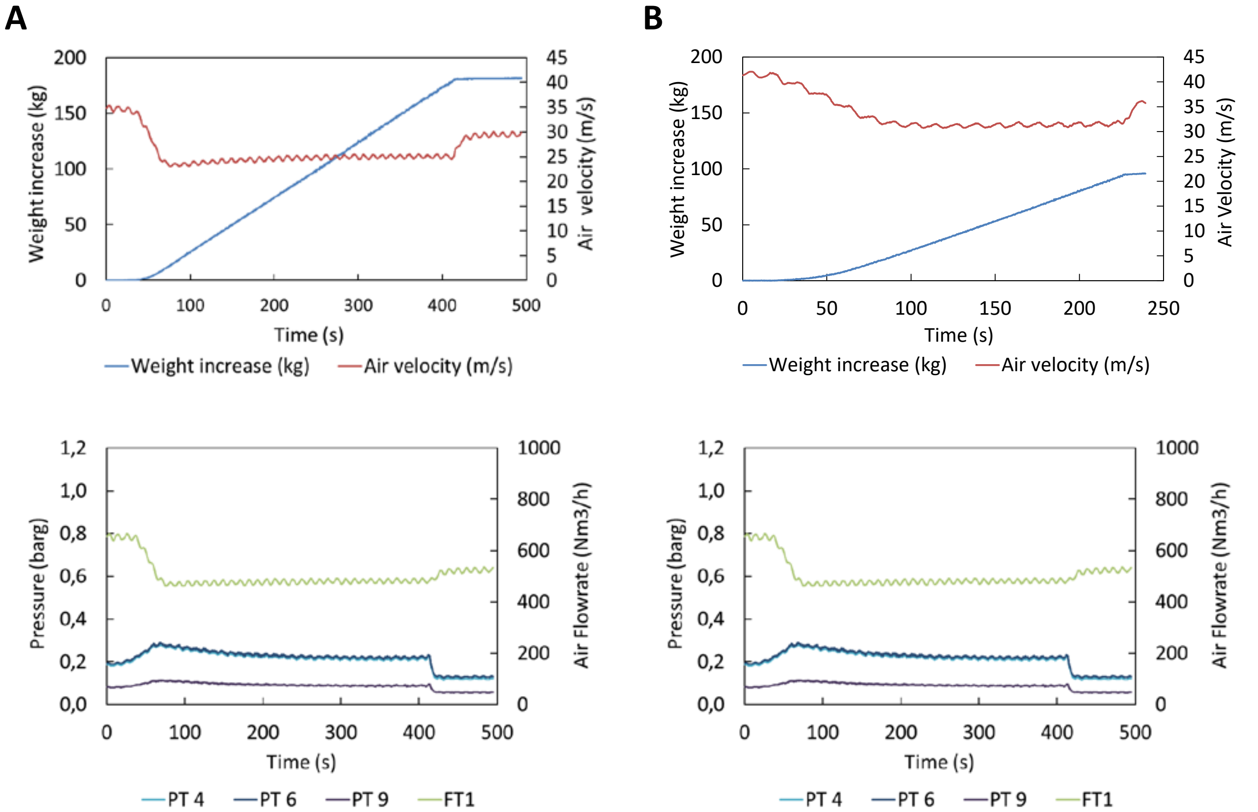


Figure 14: The first (A) and the second test (B): the top figures showing the air velocity and the weight increase by pellets accumulated in the receiving tank and the bottom figures showing the air flowrate and the pressure signals across the bend (PT4,6 and 9 are pressure transducers and FT1 is a flowmeter).

After the first two rounds, the conditions (pressure and flowrate) became less and less stable, resulting in continuous decline of air flowrate and air velocity (as low as 10 m/s at the end of the seventh round), increase in line pressure and pressure drop across the bend and higher fluctuations in the signal readings (Figure 15). It is also apparent that the amount of pellets transported into the receiving tank decreased from round to round, starting from about 180 kg in the first round and getting to 140 kg of pellets in the seventh round. After discharging the pellets from the receiving tank, it became obvious that the pellets underwent a severe degradation during the tests (Figure 16), and it was decided to fill the feeding tank with a new batch of fresh pellet material. At this point, the bend should have been detached to check the conditions (deposits) inside the pipeline. Because of concern that seven rounds were not long enough duration to create erosion that could be measured, it was decided to fill the feeding tank with a new batch of fresh pellets a repeat the procedure with additional seven rounds.

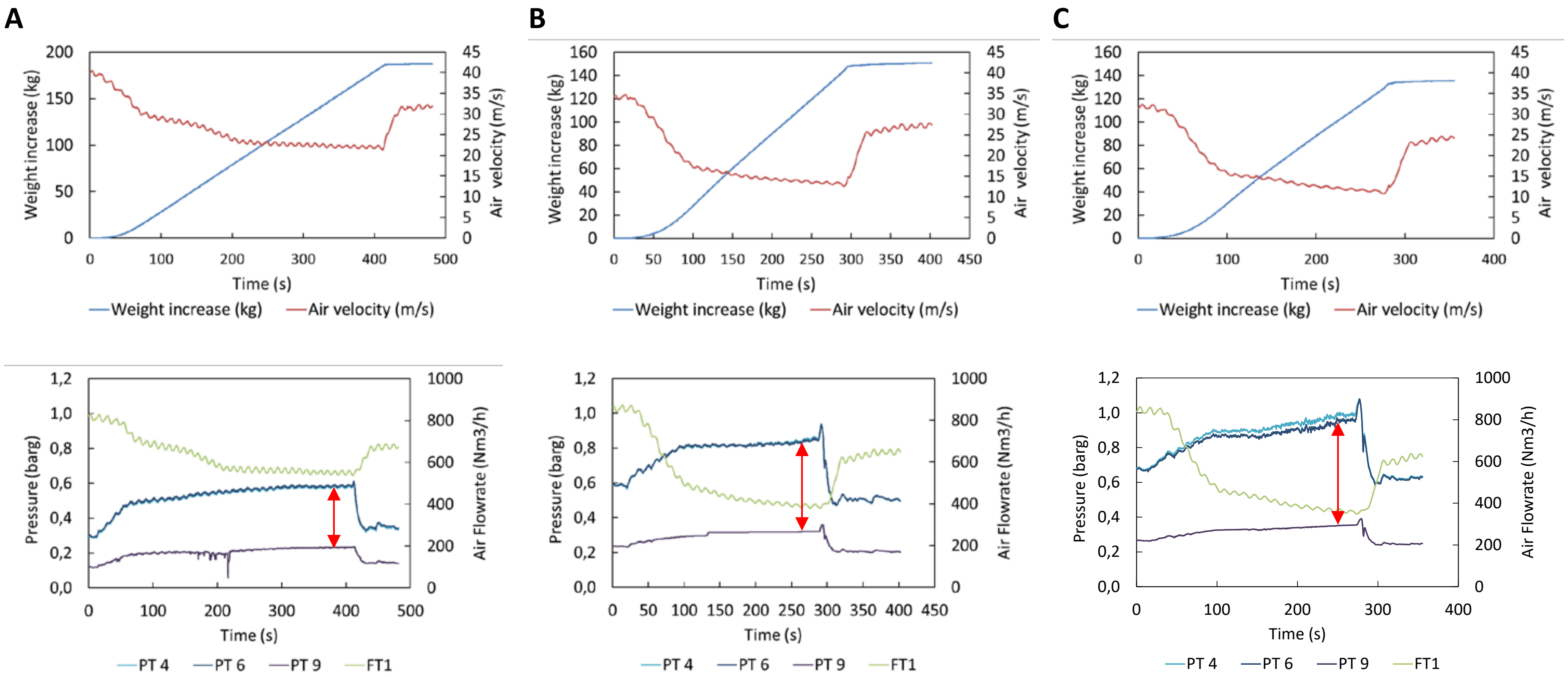


Figure 15: The third (A), fifth (B) and the seventh test (C): the top figures showing the air velocity and the weight increase by pellets accumulated in the receiving tank and the bottom figures showing the air flowrate and the pressure signals across the bend (PT4,6 and 9 are pressure transducers and FT1 is a flowmeter).

Figure 16: Degraded pellets after seven rounds of transport in the pneumatic conveying rig, the size of the pellets is reduced, and pellet debris are apparent.

Figure 17 shows the signal readings from the second, third, fifth and seventh test carried out with the new batch. The conditions were from the beginning unstable with continuous decline in air velocity and significant fluctuations in the last seventh round.

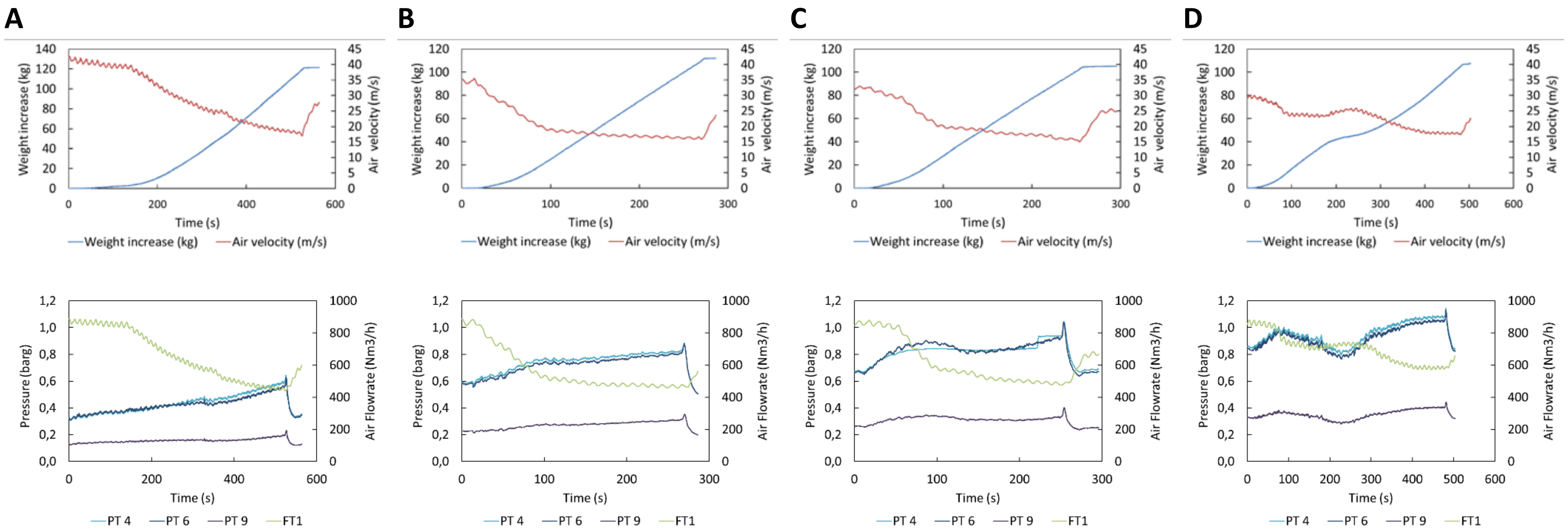


Figure 17: The second (A), third (B), fifth (C) and the seventh test (D): the top figures showing the air velocity and the weight increase by pellets accumulated in the receiving tank and the bottom figures showing the air flowrate and the pressure signals across the bend (PT4,6 and 9 are pressure transducers and FT1 is a flowmeter).

After the last round, the bend piece was detached from the pipeline, which revealed significant amounts of oily dust deposit inside the lines and in fact, the whole pneumatic conveying system (Figure 18). Such large amount of deposit inside the lines was unexpected, though it explained the unstable conditions and the material loss in the receiving tank during the tests.

Figure 18: Oily dust deposit inside the pipeline after erosion tests.

The significant degree of pellet degradation during erosion tests was attributed to combined effects of high velocity (especially during the very first two tests) and the number of bends that the pellets had to pass through during the tests. With four 90° bends in the conveying set-up and recirculating the pellet material between the feeding and the receiving tank, the pellets had to pass in total 28 times through a bend (in seven rounds). This has been detrimental to the pellets and resulted in severe breakage of pellets and oil seepage. From this, it can be concluded that the current pilot-scale set-up of the conveying line is not suitable for the study of erosion by fish feed pellets at conveying velocities above 20 m/s as this will result in pellet degradation. To study erosion at high velocities would require a change of the set-up, making it more relevant to the set-up of feeding pipes typically used at fish farms (longer straight sections and lower number of bends). However, this has not been possible under the current study.

Despite the pellet degradation at high velocity during erosion tests, observations made during the pressure tests i indicate that the pellets withstand the transport in the pilot-scale set-up at low air velocity. It is important to note that the duration of the pressure tests was also shorter compared to erosion tests, resulting in only minor pellet degradation as opposed to erosion tests.

Erosion by weight difference

Samples of the oily dust deposit from inside the bend were taken. The bend was then cleaned as described in Section b.iii and the used cleaning solution was also saved for later analysis of NMPs in WP2. After cleaning the bend, the weight of the bend was determined. The weight measurement is presented in Table 2.

Table 2: Weight measurement of cleaned bend piece before and after tests.

Weight before tests (kg)	Weight after tests (kg)	Weight difference (kg)

9.547	9.450	0.097

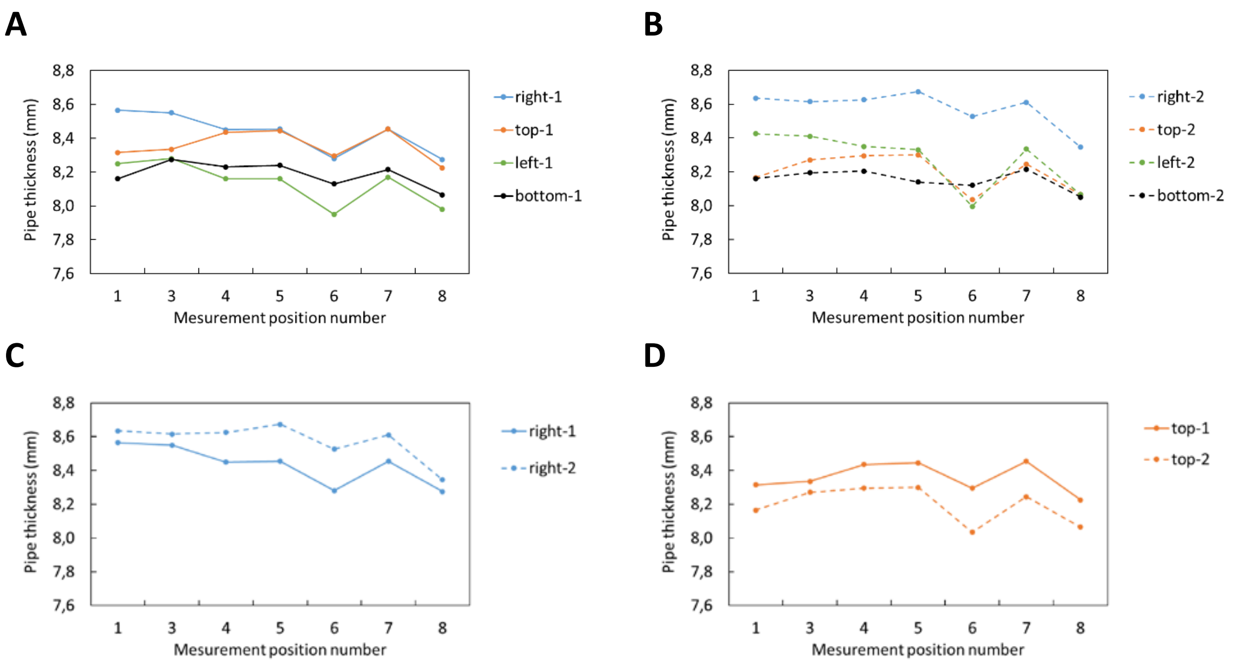
The total transport time during the tests was about 70 min and during this time, the total of 1966 kg of pellets was transported. With a bend length of 2.94 m, the amount of erosion was estimated to 0.028 kg/meter/hour. This is surprisingly a high degree of erosion, which is significantly higher than the reported values by previous studies [1], [3], [5]. However, it is difficult to make any comparisons to previous studies as it was not possible to conduct the erosion tests in this study under controlled conditions, without the effect of pellet degradation. Also, the measurement of erosion in Table 2 is based on one set of experiments without replication.

Furthermore, no conclusions can be made with respect to erosion and the amount of microplastics potentially released from aquaculture sites, because the set-up of the conveying line in the pilot-scale rig does not represent well enough a typical set-up of the feeding lines at the aquaculture sites. The conveying distance in the pilot-scale set-up is only about 40 m while in a real fish farm, the pellets may be transported in a single line over hundreds of meters. Recirculating the pellets between the feeding and the receiving tank seven times corresponds to the total distance of about 280 m. However, the number of bends the pellets must pass is considerably higher than in a real system with longer stretches of straight sections.

Nevertheless, the study shows that under extreme conditions, with high velocities, pipe wear can be quite significant in a very short time.

Erosion by pipe thickness

Pipe thickness was determined at multiple points along the bend, as described in Section b.iv, before and after the tests when the bend was cleaned. The results are shown in Figure 19.



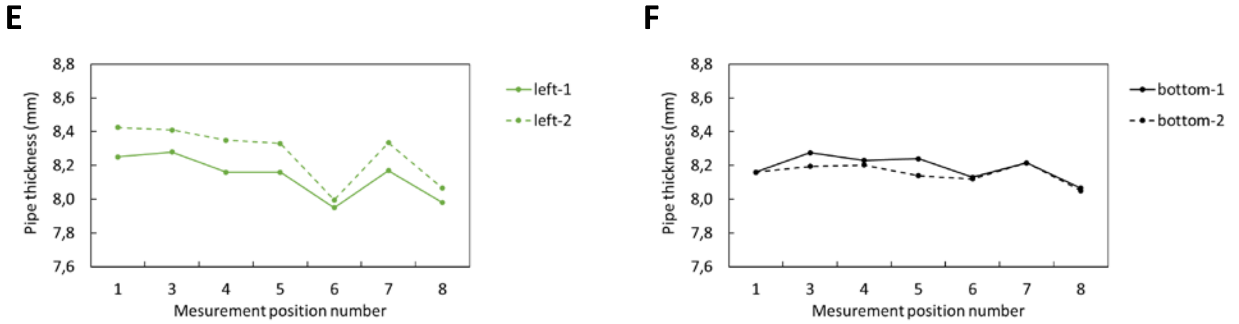


Figure 19: Pipe thickness measurements at four positions around the pipe circumference (bottom, top, right and left side): in the legend right-1, top-1, left-1 and bottom-1 are the first measurements taken before erosion tests while right-2, top-2, left-2 and bottom-2 are the second measurements taken after erosion tests.

Though the handheld Olympus pipe thickness meter has a readability of 0.01 mm, the maximum attainable absolute accuracy with proper calibration is +/- 0.10 mm. The accuracy of measurements is also reduced on rough surfaces [7]. Unfortunately, as apparent from Figure 19, the handheld pipe thickness gauge could not detect a significant difference in pipe thickness. All the measurements seem to be within the detection limit. It could be that erosion is rather uniform spreading evenly across the surface, resulting in very small difference in pipe thickness after the tests. A theoretical calculation shows that with uniform erosion across the internal pipe's surface along the length of the bend piece, the weight loss of 97 g would result in a pipe thickness reduction of 0.16 mm. Alternatively, it may be that the instrument has not been properly calibrated (calibration was not checked in between the measurements). It is also possible that the measurements have not been accurate enough due to changes in surface roughness after erosion tests. Finally, an error in measurements could have been caused by not placing the transducer at the same position due to the pipe curvature.

After measuring the weight loss and the pipe thickness, the bend piece has been cut into smaller segments and sent to WP3. More information about the more detailed surface analysis is provided in a section on WP3.

4) WP2- Quantification and characterization of eroded pipe fragments

a. Objectives

The main objective of WP2 was to characterize and quantify the amount of micro- and nanoplastic (MNP) fragments formed during the simulated distribution of fish feed pellets in a selected feeding hose performed within WP1. In WP 2, the obtained samples from different processes within the simulated fish feed supply will be analysed for plastic particles abundance, their chemical and physical properties such as polymeric composition, size and shape.

Microscopy visualization of the MP and the mass estimation of NP fragments are especially important because the plastic fragments removed from the surface of the pipe are invisible to the naked eye making it difficult to estimate the degree of microplastic contamination of the pellets in the pilot-scale pneumatic conveying tests performed in WP1.

The outcomes of the quantification and characterization exercise will support the implementation of the abrasion model performed in WP4 and will directly contribute to optimization of the feed hoses (configuration, composition) and feed delivery parameters to minimize particle generation and emissions in the aquaculture production process. Furthermore, the outcomes of WP2 will provide additional insights into the character of the MP fragments the fish are potentially exposed to during seafood production activities.

b. Materials and methods

The WP was divided in three sequential tasks aiming at: 1) Performing an accurate samples preparation to remove any potential interferent hampering the ability of the selected analytical techniques to provide reliable data; 2) Quantifying and characterizing the microns sized eroded microplastic fragments; 3) Quantifying and characterizing the nanometric sized eroded microplastic fragments.

The following test samples were obtained from the conveying pilot tests:
Pristine

- Pristine fish feed pellets for microplastics content preliminary characterization.
- Cleaning sponge for constituent polymer characterization
- Section of feeding hose for constituent polymer characterization
- Deposit directly from bend
- Pristine cleaning solution (before cleaning)
- Used cleaning solution (after cleaning)
- Feeding hose recirculated fish feed pellets
- Dust collected from filter

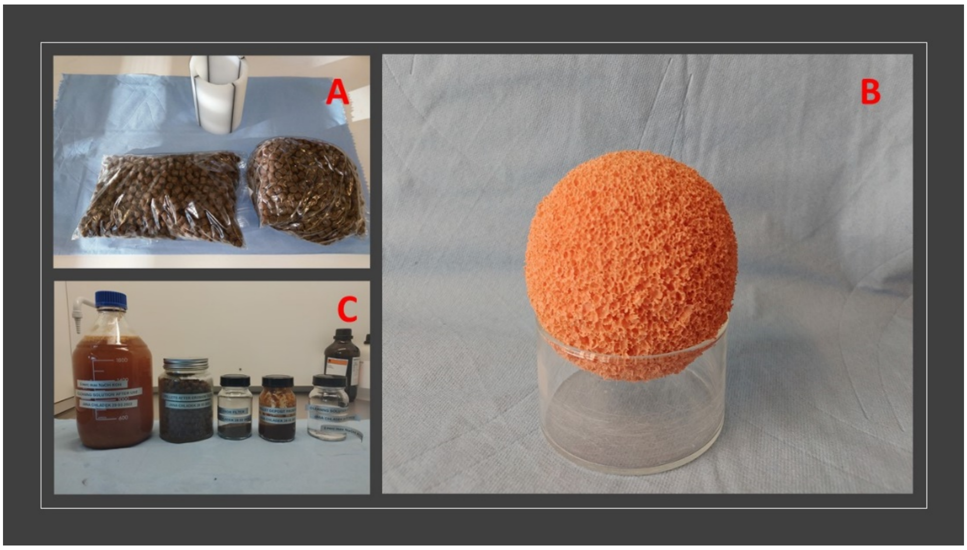


Figure 20: Testing material received form WP1. A) Pristine fish feed pellets and a sample of the feeding hose used within the convey pilot test; B) Sample of the cleaning sponge kindly provided by AKVA Group; C) samples collected from both different areas in the pellets conveying system and steps in the simulation process.

<u>Task 2.1 Samples preparation to analysis targeting fat removal, isolation, and fractionation</u>

For the optimization of the sample preparation steps the working group will initially benefit of the previous research performed within the FHF granted project "TRACKPLAST" [6]. To remove proteins and fatty compounds representing a thick surface layer in the analysed fish feed pellets (fig. 16), a multi-steps sequence of dispersants, enzymes and oxidizing treatments were tested. Different combinations of enzyme concentrations, oxidizing agent concentrations, reaction times and reaction temperatures, were tested

before an optimum protocol for removal of interfering organic compounds was identified. The protocol was used for the further sample processing. Tests on pristine and weathered pellets were conducted in 5 replicates of 50 g each. The final analysis on the solid testing material was performed on a homogenised subsample of 100 g. Combined liquid/solid samples were prior dried at constant temperature (50 C) and the obtained solid material submitted to analysis. Commercially available batches of protease, lipase and lignin oxidase for the enzymes-driven samples digestion were obtained from a professional supplier (Sigma, Darmstadt, Germany).
After combined sequential combination of enzymes incubations and strong oxidative treatments the resulting digestates were density separated and the floating particles size fractionated into two dimensional classes: D1 > 300 µm and 300 > D2 > 10 µm while the filtered digestates were collected for the nanoplastics fraction characterization (Task 3).

Task 2.2 – Quantification and characterization of eroded microplastic fragments

The assessment of the micron-sized plastic particles (MPs) will be characterized by a vibrational spectroscopy oriented analytical method. D1 represents a size fraction with large fragments and will be quantified by Attenuated Total Reflectance (ATR-FTIR).
The fraction of the finer particles still in the microns size range (D2) will be analysed through a µ-FTIR imaging microscope. Obtained spectra are be compared with commercially and non-commercially available reference infrared spectra to characterize the occurrence and the chemicals composition of plastic polymers and other organic material fragments in the sample. The combination of the introduced techniques allows to gain accurate knowledge about shape and size of plastic particles in the range of > 300 µm to 10 µm as well as the total plastic fragments quantity and the relative abundance of each of the most environmentally relevant polymer types in the investigated samples. The results from this Task will feed directly into optimising the configuration and composition of the feeding system (WP1), as well as model development (WP4).

Task 2.3 – Qualification of eroded nanoplastic fragments
This task comprised two parts:

1) identify one or more polymer-specific chemical markers from the HDPE tube material using a non-target analytical chemical approach
2) determine if the targeted HPDE polymer chemical marker(s) is presence in the nanoplastic fraction

A screening for the presence of polymer-specific markers in the plastic feeding tube material was performed by analyzing and comparing solvent extracts and aqueous leachates derived from the polymer tube. A non-target screening employing full scan GC-MS analyses compared the extract to the leachate in the quest for polymer-specific markers that would not leach to the aquatic matrix. Here we were looking to identify chemicals present in the plastic tube material that did not leach into water. Briefly, 6 x 500 mg of the HDPE tubing was sampled and cut to thin pieces. Half of the material was extracted using 4 mL dichloromethane (DCM) with 20 min ultrasonication to generate a solvent extract. The other half of the tube material was placed in glass bottles containing with 1 L tap water and allowed to leach for 7 days at room temperature in the dark. The chemicals present in the resulting aqueous leachate were solvent extracted into DCM (120 mL in 3 portions) and up-concentrated using a TurboVap®. The final volume of the extracted leachate sample was adjusted to 1 mL. Both the solvent and the leachate samples were spiked with surrogate internal standards (Phenol-*d6* 25.2 µg, *p*-Cresol-*d8* 1.1 µg, n-4-propylphenol-*d12* 1.3 µg, Naphthalene-*d8* 1µg, Phenanthrene-*d10* 0.5 µg, Chrysene-*d12* 0.5 µg, and Perylene-*d12* 0.5 µg) prior to extraction. Recovery standards (Fluorene-*d10* 1 µg *and* Acenaphthene-*d10* 1 µg) were added immediately prior to analyses. The chemicals identified as suitable polymer-specific markers (see results section) were used to develop a target analyte analysis method based on the retention times and diagnostic fragment ion masses from the mass spectra.

The sub-20 µm particle fractions collected from the MP digest in Task 2.1 were extracted and analyzed for the presence of the chosen polymer-specific markers identified from the pre-study described above. The received samples were digests of (i) neat pellets, (ii) pellets that had been run through PE feeding tube, (iii) the pellet deposit in the PE feeding tube, and (iv) the cleaning solution after use. The extraction procedure was similar to the aqueous leachate extraction. Surrogate internal standards were added prior to extraction (Phenol-*d6* 25.2 µg, *p*-Cresol-*d8* 1.1 µg, n-4-propylphenol-*d12* 1.3 µg, Naphthalene-*d8* 1µg, Phenanthrene-*d10* 0.5 µg, Chrysene-*d12* 0.5 µg, and Perylene-*d12* 0.5 µg) and the samples were extracted using DCM (120 mL in 3 fractions). The extracts were ultrasonicated to liberate phenolic compounds from the lipid rich samples and frozen at -20^{o}C to remove lipids, this was repeated three times. The liquid phase was then extracted with 30 mL dichloromethane and filtered through GF/F-filter. The samples were evaporated using a Turbovap. The lipid rich extract was diluted to a 10 mL and a 900 µL aliquot was taken. Next, 100 µL recovery standard (Fluorene-*d10* 1 µg *and* Acenaphthene-*d10* 1 µg) was added to the 900 µL aliquot to give a final sample volume of 1 mL. The extracts were then analysed by the targeted (SIM) GC-MS method developed from the first part of the study.

Analysis of the extracts was performed using an Agilent 7890A GC gas chromatograph (GC) coupled to an Agilent 5975C mass spectrometer. Samples (1 µL) were introduced at 250°C in pulsed splitless mode. Separation was achieved using a Zebron ZB-1MS column (30 m length, 0.25 µm film thickness and 0.25 mm internal diameter). The carrier gas was helium at a constant flow of 1.1 mL/min. The column oven temperature was programmed at 40 °C (2 min), ramped by 10 °C/min until 320 °C (10 min hold). The transfer line temperature was 300°C, the ion source temperature 230 °C and the quadrupole temperature 150 °C. The ion source was operated at 70 eV, with a solvent delay of 8 minutes.

c. Results

Task 2.1

This task aims at extracting MPs from the investigated samples by applying a gentle and efficient purification step prior to chemical identification allowing for a quantitative analysis. The main interferents for a reliable quantification are a complex mixture of proteins and fats (natural esters of glycerol, as well as fatty acids) that may trap and aggregate MPs. The fats in the dry feed materials present a problem for extraction and purification of samples, i.e., for the separation of the MP fragments from the remaining medium. These are factors that can reduce the efficiency of the extraction process, as well as interfere with the chemical analysis and quantification process, causing an increase in the background signal and reduce the signal-to noise ratio.
The sample's preparation procedure was set to separate the fats and lipids fraction from the solid matrices within the first steps of the procedure allowing to process separately the liquid fats-enriched from the solid protein-based fraction. The results of the optimization are seen in Figure 21, showing the final flow chart for sample preparation of the solid samples.

The initial treatment with 10% Tween-20 in 10% KoH helped the lipids/fats fraction to float in the beaker. The supernatant was collected and the sedimented solid material submitted to a sequence of enzymes to remove protein, carbohydrates and small quantities of lignin-like and residual lipids. The obtained digestate was further submitted to strong oxidation treatment to eliminate any residues of organic matter. The obtained sample was combined with the floating fraction and filtered through a certified 10 µm mesh size stainless steel filter. Filtrates were submitted to analysis according to task 2.3 while filter trapped material was submitted to density flotation in a dense solution of KBr. After 5 days of flotation the supernatant material was collected, filtered and resuspended in a fixed volume (1 mL) solution of ethanol/water. Homogeneous aliquots (200 µl) of this solution were deposited on ZnSe window for IR scanning.

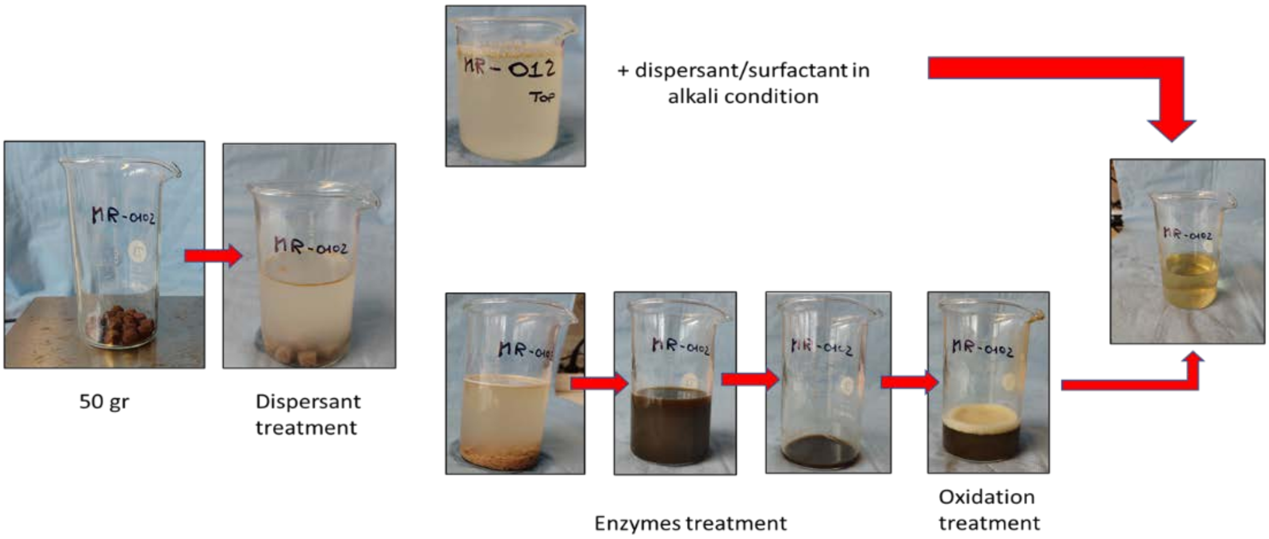


Figure 21: **ATR-FTIR Sample acquisition (left) and IR profile (right) acquired during the characterization of the cleaning sponge and identification exercise by comparison with Hummel IR polymers reference database.**

Task 2.2

The ATR-FTIR analysis submitted to both a fragment of the cleaning sponge and a section of the feeding pipe identified Polyethylene (PE) as the constituent polymer in both cases (fig. 22).

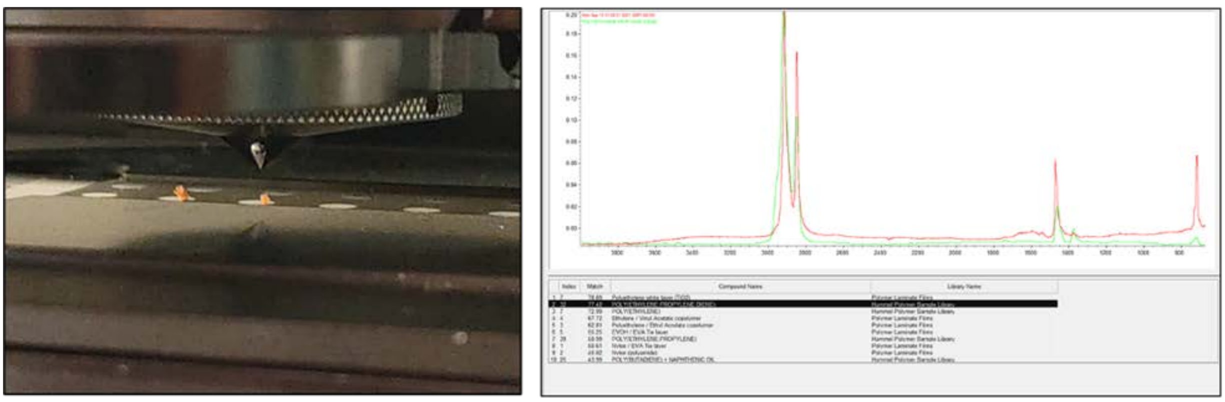


Figure 22: **ATR-FTIR Sample acquisition (left) and IR profile (right) acquired during the characterization of the cleaning sponge and identification exercise by comparison with Hummel IR polymers reference database.**

For the preliminary characterization of the microplastics content in the fish feed pellet used in the conveying test, the samples were prepared for the analysis by applying the sample preparation procedure developed within the task 2.1.
Concentrates of the digested samples were deposited on a ZnSe infrared compatible window and scanned by µ-FTIR imaging system. The resulting heat maps are analyzed by a post-acquisition algorithm able to compare pixel by pixel the obtained IR spectra with those recorded within built-up IR standards reference library. Relevant matches are plotted on a bidimensional map pointing out the shape and the polymer type (fig. 23).

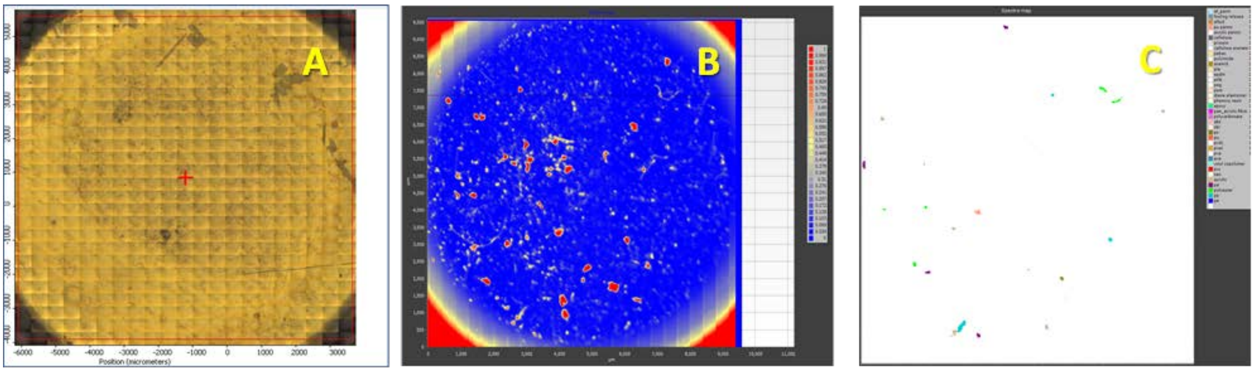


Figure 23: **µ-FTIR imaging on samples of pristine fish feed pellet. (A) true colour picture of a sample deposited on a ZnSe window; (B) associated heat map; (C) localization of plastics fragments in the scanned window.**

The results point out a total number of plastic fragments ranging from 63 to 144 MPs/100 gr DW in the analyzed replicates. Polyethylene, Polypropylene, Nylon/Polyamide and Polypropylene Terephthalate the most recurring polymer types in the analyzed samples. The fragments' dimensions ranged from 20 µm to 105 µm being 48 µm the median size range. Overall, the particle's from was the dominant shape (64%) respect to the fiber one (36%).

In a similar way were analyzed also the samples collected from different steps during the conveying simulation experiment. Overall, Polyethylene was the dominant polymer type in the microplastics composition which abundance spanned from 89 % in the reference cleaning solution to 97% observed in the deposits from belt samples (fig. 24). Polystyrene (1-5%) and Polyamide (1-3%) were respectively the second and the third most accounted polymer types in the investigated samples. Low levels of Polyethylene Terephthalate, Ethylene Vinyl Acetate, Polypropylene, Poly Ethyl Sulfone, Polyurethane and Polystyrene were also recorded but not systematically in all investigated samples.

![SINTEF logo]

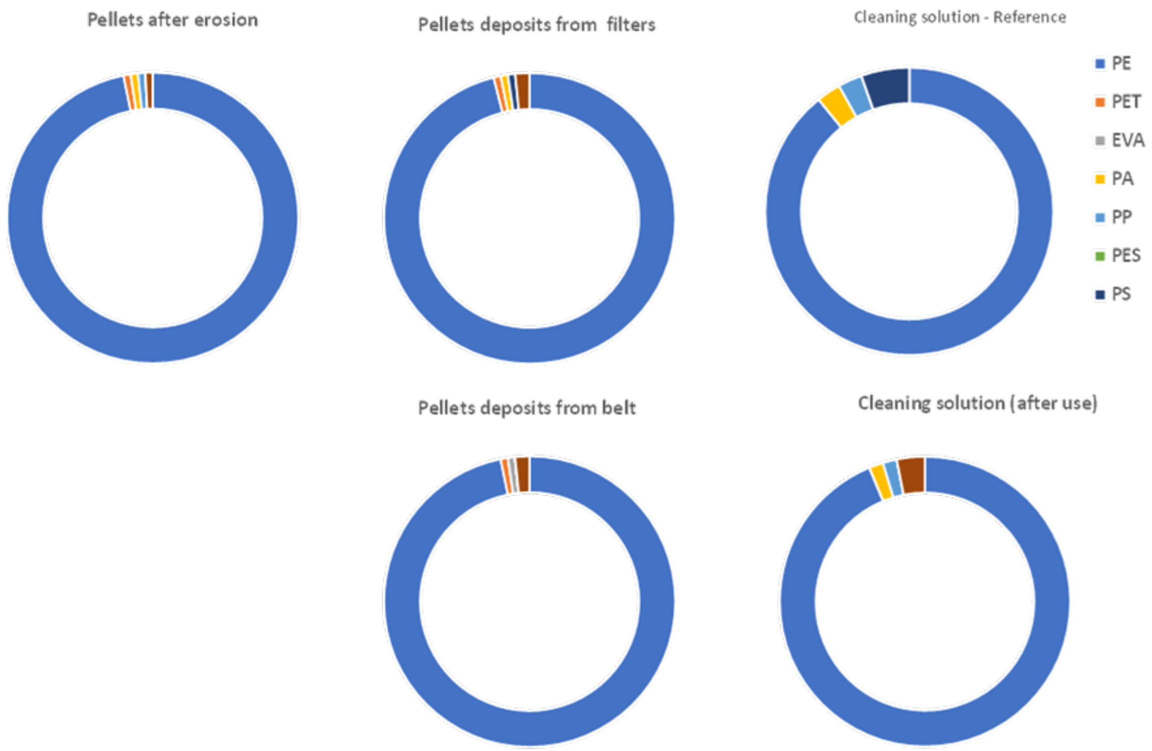


Figure 24: **Relative polymers composition in the analysed samples from the convey simulation test.**

The total number of microplastics in the samples varied significantly with the cleaning solution before its use (reference) showing 7 MPs/L and a significant increment after use (196 MPs/L). On a similar way, the semi-solid and solid samples showed significant differences with lower levels observed in the pellet deposits on belt (23 MPs/g DW) and considerably higher amounts in the pellet's surfaces after the erosion processes (278 MPs/g DW, fig 25).

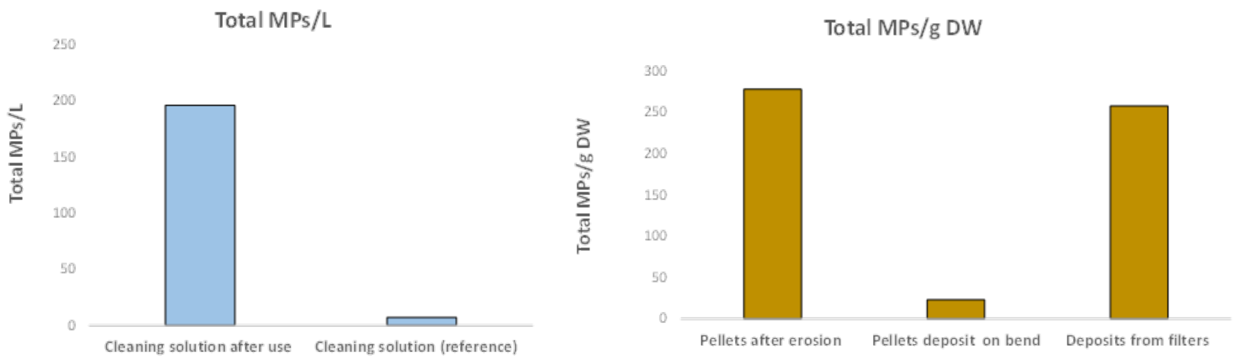


Figure 25: **Total amount of MPs identified in the analysed samples from the convey system experiment.**

Furthermore, the particle size distribution analysis of the investigated samples from the convey system experiment showed informative results. If the material deposited in the filter showed a size distribution toward the smaller particles. An opposite distribution prioritizing larger fragments is observed in in the pellet

material after erosion. On the other hand, the liquid cleaning solution point out a more even fragment's size distribution.
Overall, the fats layer coating occurring in the inner section of the feeding hoses during standard fish feed pellet distribution (fig.18) may play an important role in explaining the observed plastic fragments size distribution in the addressed samples.

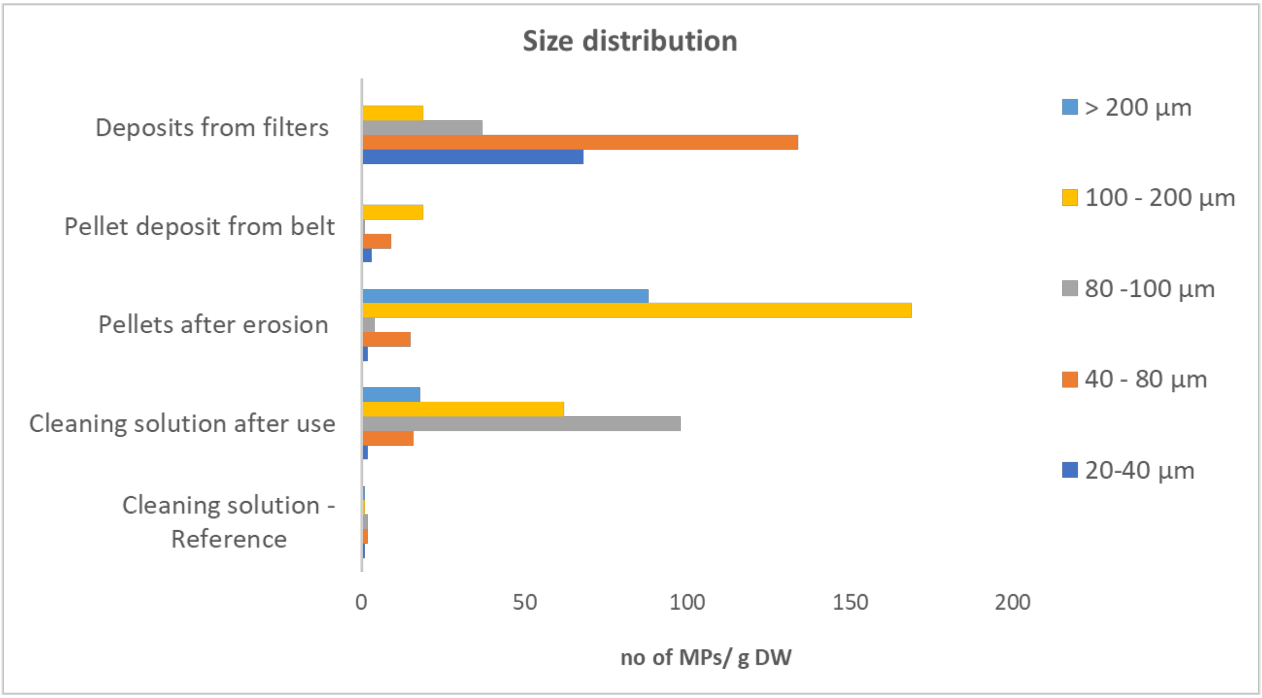


Figure 26: **Relative size distribution of plastic fragments in the analysed samples from the convey system experiment.**

Task 2.3

The characterization of the HDPE tube using a non-target screening approach resulted in three suspect polymer-specific marker chemicals being identified: dibutyl phthalate, 2,4-bis(1-methyl-1-phenylethyl)-phenol, and 2,4-Bis(dimethylbenzyl)-6-t-butylphenol with 278, 330, and 386 *m/z* as respective characteristic ions. These chemicals were detected only in the solvent extract and not in the leachate sample, indicating that they are specific to the plastic tubing and do not readily leach. As such, they a potentially good chemical markers for the detecting the presence of nanoplastic in a sample. There were several chemicals detected in the leachate, however, further identification and characterisation of these chemicals were outside the scope of this study. The solvent extract and the leachates were then re-analysed with the targeted GC-MS method (using single-ion-monitoring) for validation of these chemicals and their presence in the solvent extract only, see Figure 27.

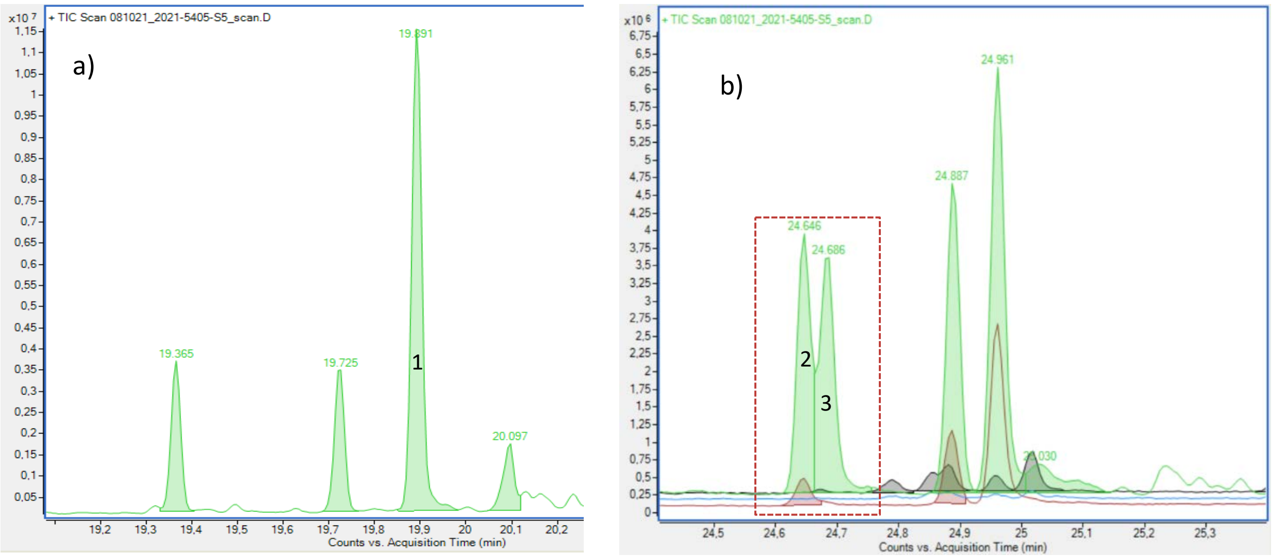


Figure 27: Chromatogram showing suspect markers, I.e., peaks present in solvent extract only. a) Suspect marker 1 with RT 19.89 min. b) Suspect markers 2 and 3 showing with RT 24.65 and 24.67 min. In figure b) there were some low-intensity peaks present in the solvent extract (green), leachate (black), solvent extract procedural blank (red), and leachate procedural blank (blue), these are overlaid in b)to show that there is little to no interference with the suspect markers 2 and 3.

The chromatograms from the target SIM analysis of the laboratory blank are shown in Figure 28, with each chromatogram showing the response for one of the identified diagnostic fragment ions from the 3 candidate chemical markers. The results show that there is low signal response for all 3 target analytes, confirming there is little to no background contribution from these chemicals from sources within the laboratory. Therefore, contamination of the real samples can be assumed to be negligible.

The SIM chromatograms for the diagnostic ions (278 *m/z*, 330 *m/z*, and 386 *m/z*) for the three candidate chemicals markers for each of the sample types (cleaning solvent after use, pellet deposit from bend, pellets after erosion, and pellets) are presented in Figures 29-30, respectively. The results show no indication of any of the targeted analytes in any of the samples. Although there are signals/responses in most of the chromatograms for the selected ions 278 *m/z*, 330 *m/z*, and 386 *m/z*, these are not present at the correct chromatographic retention time of dibutyl phthalate (19.89 min), 2,4-Bis(1-methyl-1-phenylethyl)-phenol (24.65 min) and 2,4-Bis (dimethylbenzyl)-6-t-butylphenol (24.67 min).

Based on the results, we can conclude that there is no particulate material present in the samples below 10 µm. However, it is important to note that there are limitations with this approach, the primary one being that it is impossible to know without further study and the use of reference chemicals for each of the target chemical markers how sensitive the approach actually is. It cannot, therefore, be concluded that there is no particulate material present below 10 µm, but rather that any material present was below the detection limit of the method developed and implemented within the MICRORED project. Furthermore, the sample preparation procedure for both the enzymatic digest and extraction may have caused degradation and evaporative losses, respectively, of the target compounds. This would also need verification using pure standards of each chemical. It is also important to note that this approach was developed because there are current no reliable methods available for the extraction, identification and quantification of nanoplastic from complex environmental matrices. Such methods are currently under development and therefore the issue of nanoplastic emissions should be re-visited in the future when the methods are implementable.

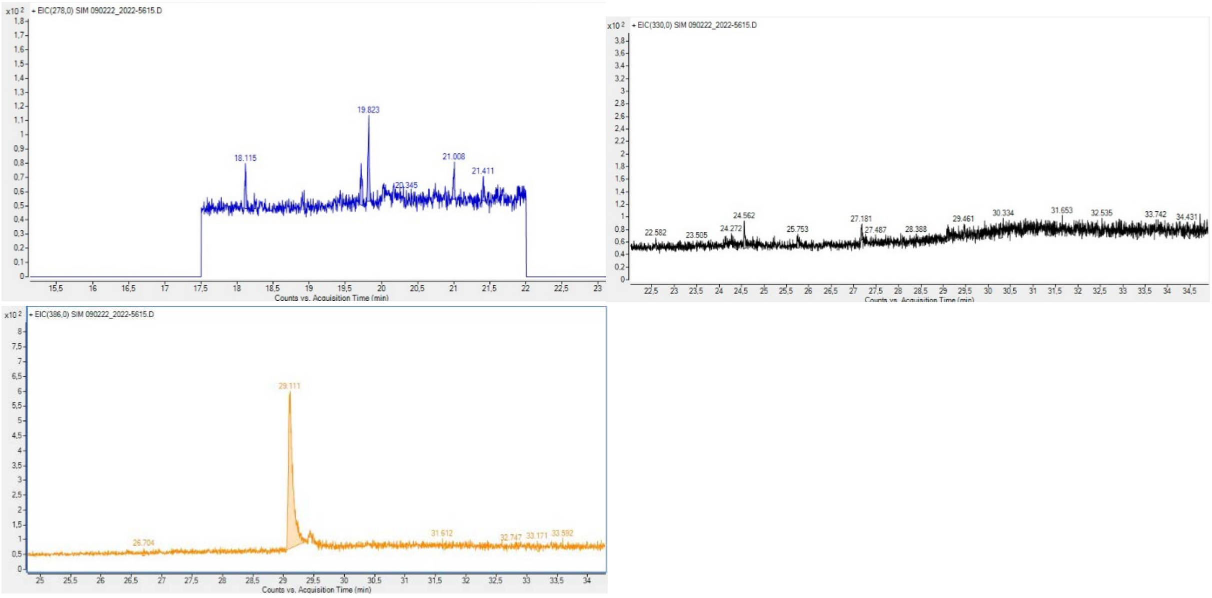


Figure 28: GC-MS SIM chromatograms of the laboratory blank sample showing responses of the diagnostic ions for the three candidate chemical markers; 278 *m/z* (top left, blue), 330 *m/z* (top right, black), and 386 *m/z* (bottom, orange).

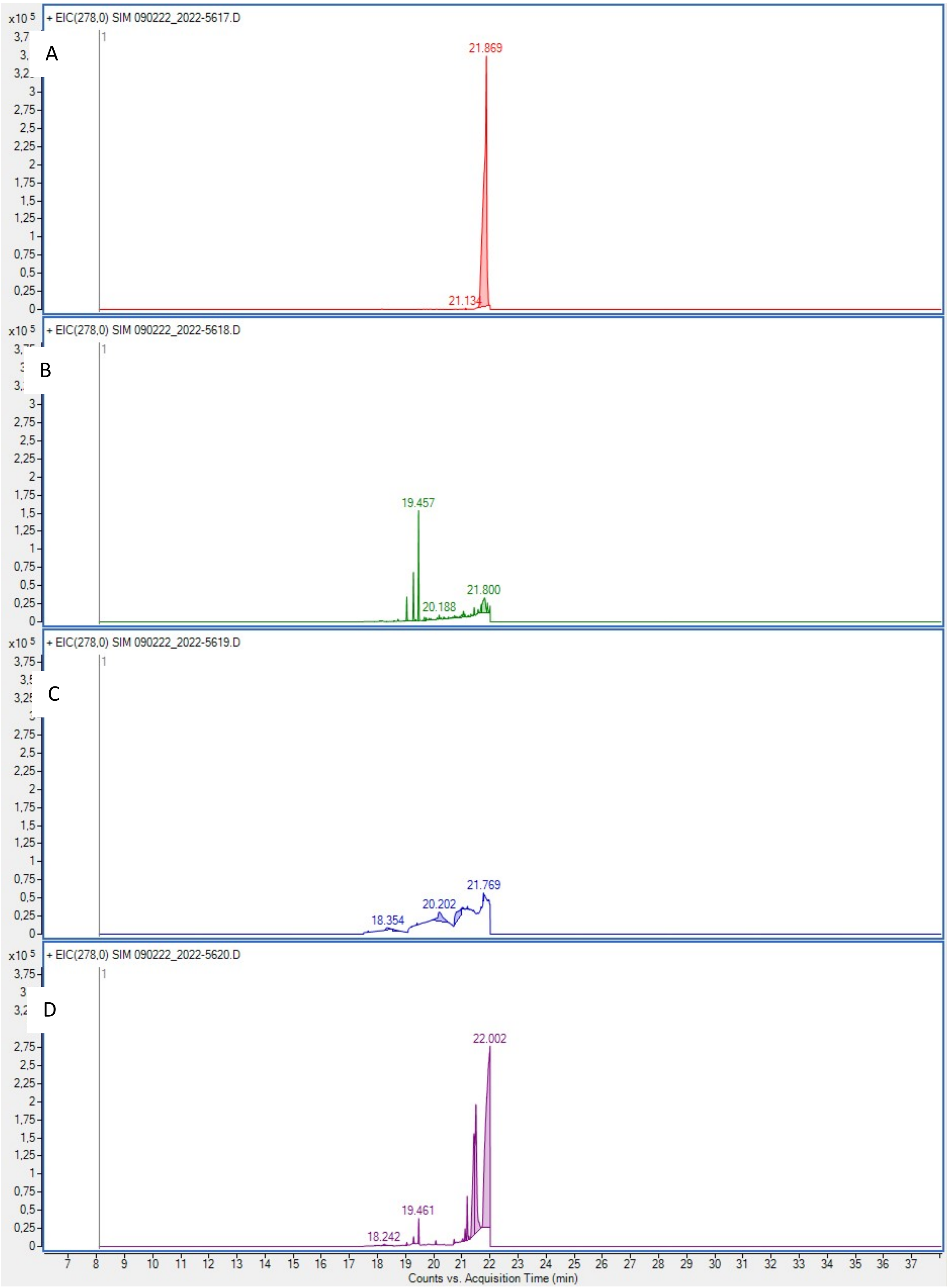


Figure 29: GC-MS SIM chromatograms looking for the presence of marker chemical #1 (dibutyl phthalate) with diagnostic ion 278 *m/z* and a retention time of 19.89 min in solvent extracts of (A) cleaning solvent after use, (B) pellet deposit from bend, (C) pellets after erosion, and (D) pellets.

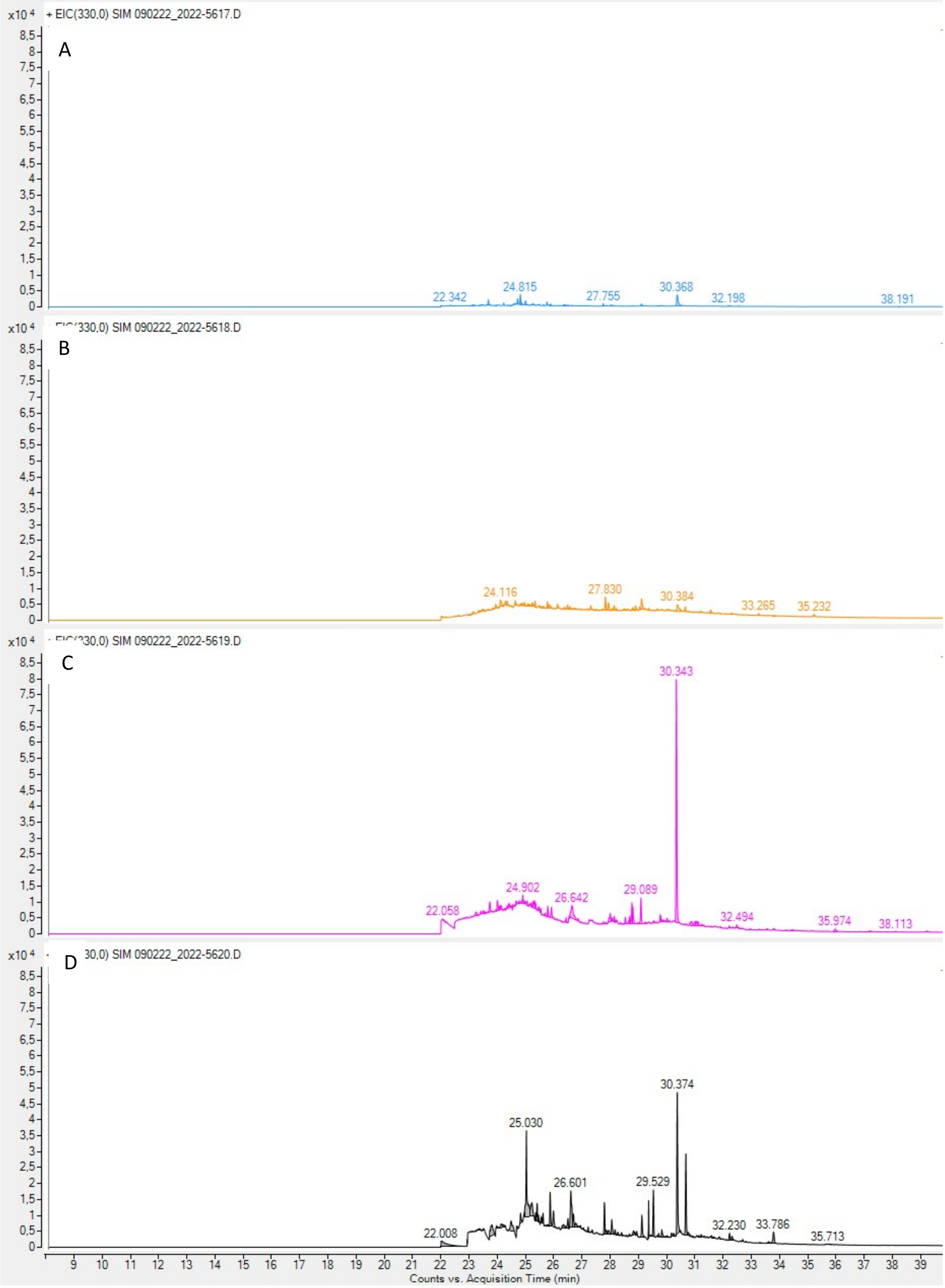


Figure 30: GC-MS SIM chromatograms looking for the presence of marker chemical #2 (2,4-bis(1-methyl-1-phenylethyl)-phenol) with diagnostic ion 330 *m/z* and a retention time of 24.65 min in solvent extracts of (A) cleaning solvent after use, (B) pellet deposit from bend, (C) pellets after erosion, and (D) pellets.

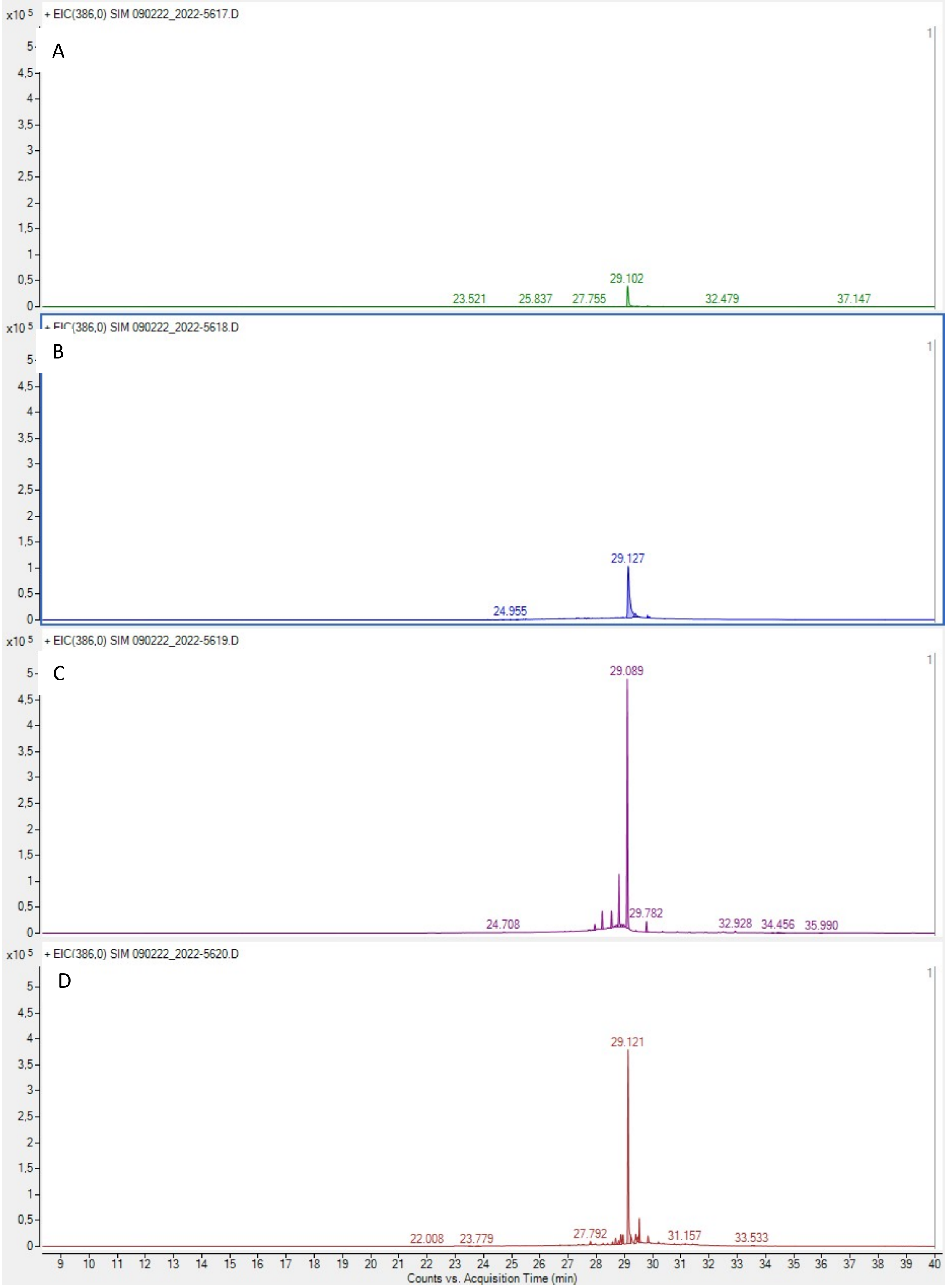


Figure 31: GC-MS SIM chromatograms looking for the presence of marker chemical #3 (2,4-Bis(dimethylbenzyl)-6-t-butylphenol) with diagnostic ion 386 *m/z* and a retention time of 24.67 min in solvent extracts of (A) cleaning solvent after use, (B) pellet deposit from bend, (C) pellets after erosion, and (D) pellets.

5) WP3- Morphological investigation of erosion on pipeline surface and erosion evolution of pipelines

a. Objectives

In WP3, it was proposed to carry out the morphological investigation of the eroded pipeline obtained from WP1, the objectives for WP3 are

1. To visualize the erosion pattern of the pipeline under selected conveying conditions in WP1
2. To provide information on the character of the surface damage on the pipe wall surface

The knowledge achieved in WP3 will provide deeper understanding on how the surface erodes when a pellet collides with the surface at high velocity at different impact angles is needed for identification of the wear erosion mechanisms, which will be utilized to optimizing the fish feeding systems.

b. Materials and methods

i. Preparation of virgin and eroded pipe segments

As described in Section 3.2.3, the bend section was removed from the model conveying system and cleaned before being cut into smaller segments and sent to SINTEF Polymer and composition materials group in Oslo. The segments received from WP1 was aligned, and its corresponding position in the conveying system is shown in the schematic figure below (Figure 32). The length of the detachable bend part was 2.94 m, and it was cut into 8 smaller segments, each has a length between 30-40 cm.

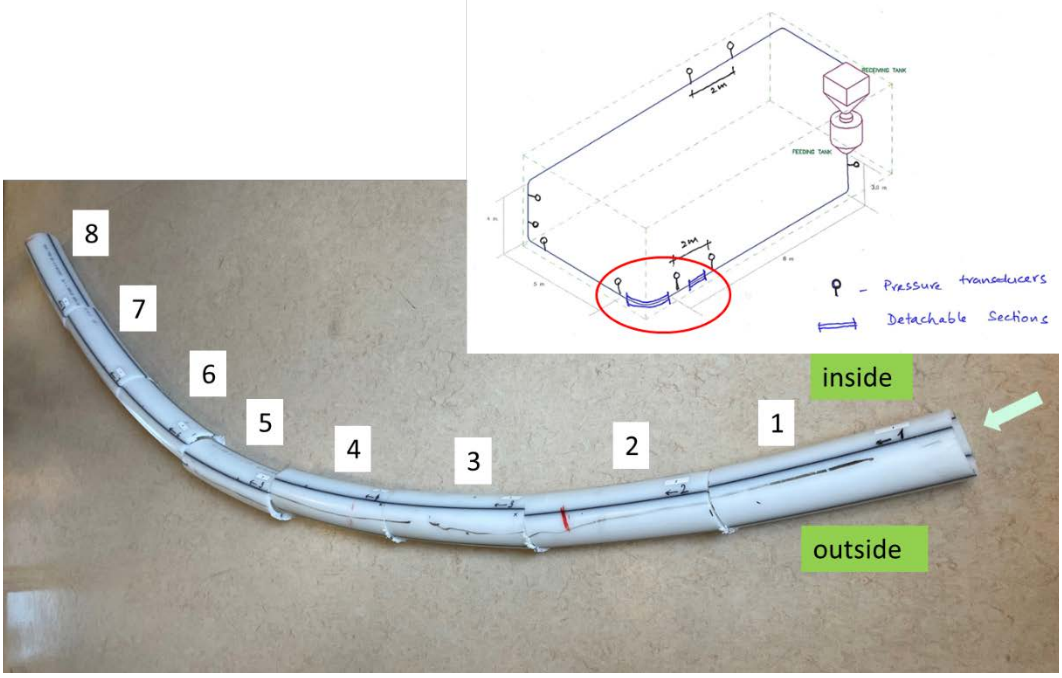


Figure 32: Schematic of the conveying system

Each segment was then cut open along the pipeline following the two black ridges located on the top and bottom of the pipe, and the obtained two parts were noted as "outside" and "inside" parts, correlating to the turn of the bend, respectively.

In addition, one piece of virgin pipe with a length of 30 cm was received as reference sample. It was also cut into two along the black ridges.

ii. Morphology investigation

Visual observations and optical images

The cut-opened pipe segments were observed visually to get an initial idea of the erosion situation. To document the erosion, optical images were taken from both horizontal and vertical directions under natural light with the same setting of parameters.

White light interferometry (WLI)

After the initial examination of individual pipe segments, the surface texture and geometric dimensions of the selected segments were investigated by a white light interferometer (Veeco Wyko NT9800, Bruker). WLI is a non-contact optical method for 3D surface measurement and inspection, which can be used to investigate the surface texture and geometric dimensions. The samples were cut from the selected positions on the pipe segments with a size of 14 x 20 mm.

c. Results

i. Comparison of inner surface appearance of the virgin and eroded pipe segments

Virgin pipe

The inner wall of the virgin pipe is semi-glossy, and the surface is smooth. When rotating under the light, ridges along the extrusion direction can be observed, which are attributed to the extrusion process. The images of the inner wall of the virgin pipe are presented in Figure 33.

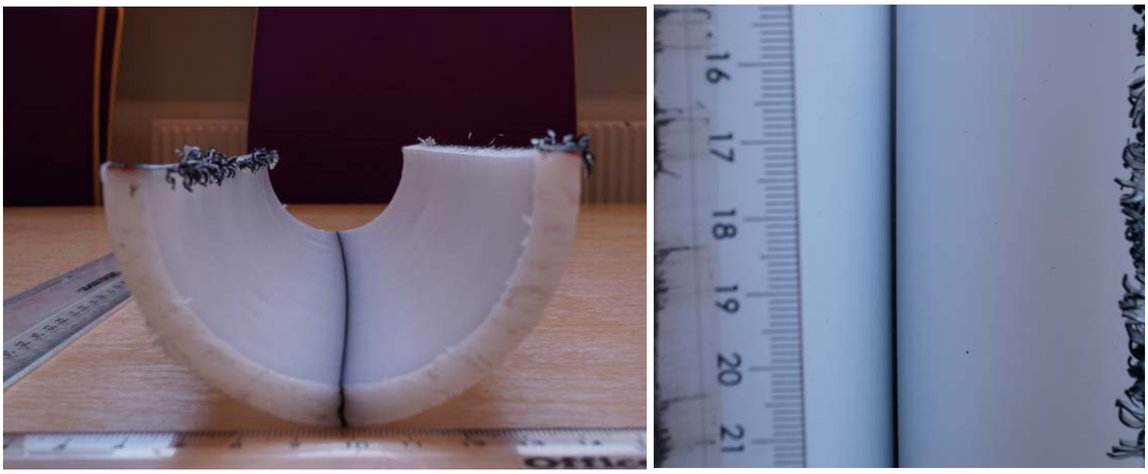


Figure 33: Optical images of virgin pipe. Left: horizontal; right: Vertical

Eroded pipe

All eroded segments are matt, indicating the erosion of inner wall with tiny dents and stubbles. It is worth noting that the erosion patterns are varying along the pipeline as well as the positions of the pipe (inside or outside of the bend). Images were taken for all 16 half-pipe segments, limited by the space, only images of Segments 1, 4 and 8 are presented here, representing the straight part (No.1), the summit of the bend (No.4) and the end of the bend (No.8), respectively.

As can be seen from the images of segment 1 (Figure 34 and Figure 35), the surface of the inner wall of pipe was eroded relatively evenly. When we move forward towards the apex of the bend, segment 4 shows significant difference in the erosion patterns between the inside and outside half. The scratching lines in the outside half are not only deeper and more remarkable, but more condensed than those in the inside half, indicating more severer erosion on the outside half of the pipe, which can be attributed to the collision and frication of massive feeding pellets at the elbow of the bend. Moving further to the downstream, at segment 8 the pipe starts straightening out. Compared to segment 4, the erosion on the outside half part of the pipe is alleviated but the inside half is more remarkable, suggesting that after the elbow, the distribution of the feeding pellets started to even out.

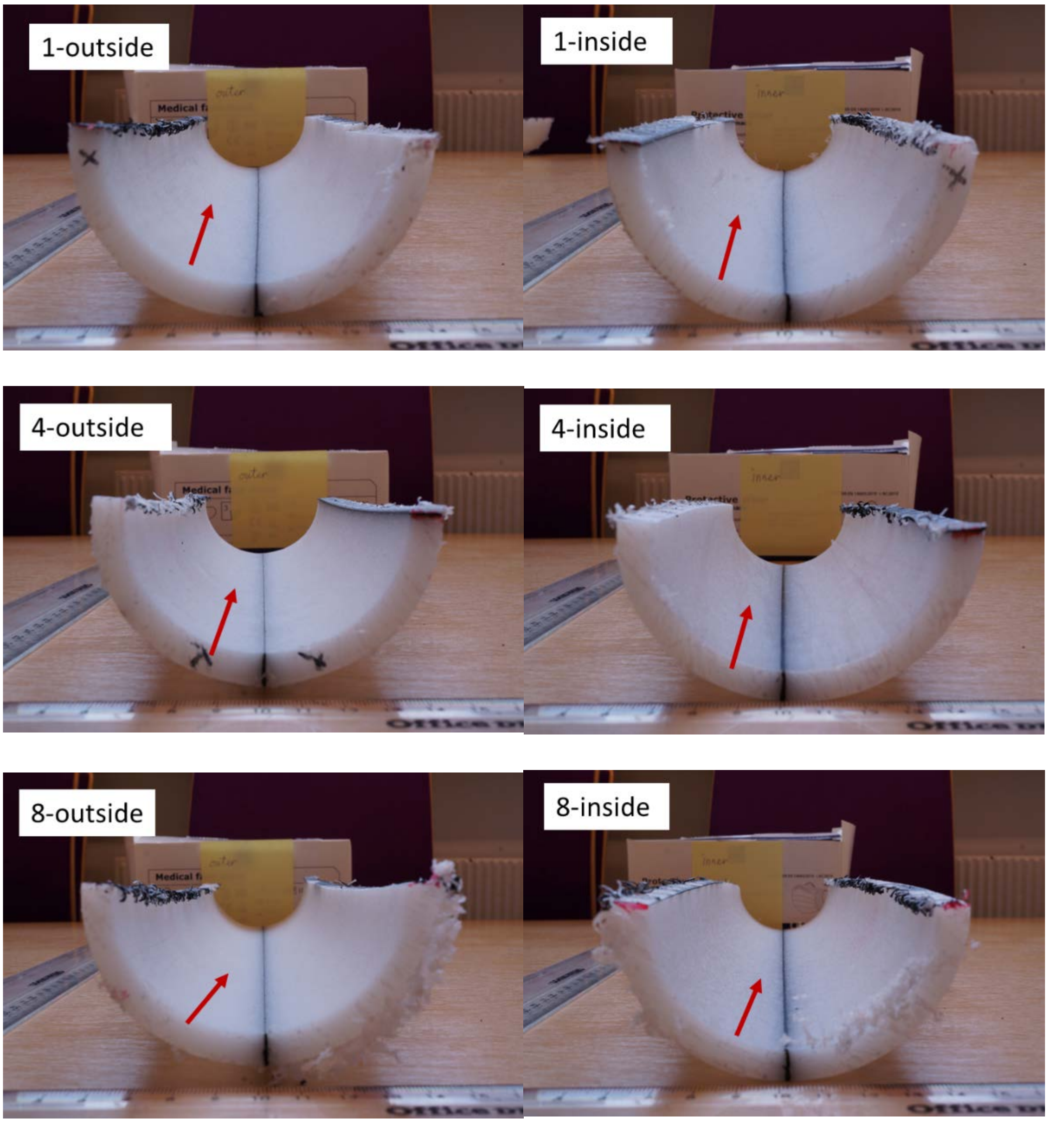


Figure 34: Optical images of individual bend segments 1, 4, and 8 taken from horizontal direction. Left: outside half pipe; Right: inside half pipe

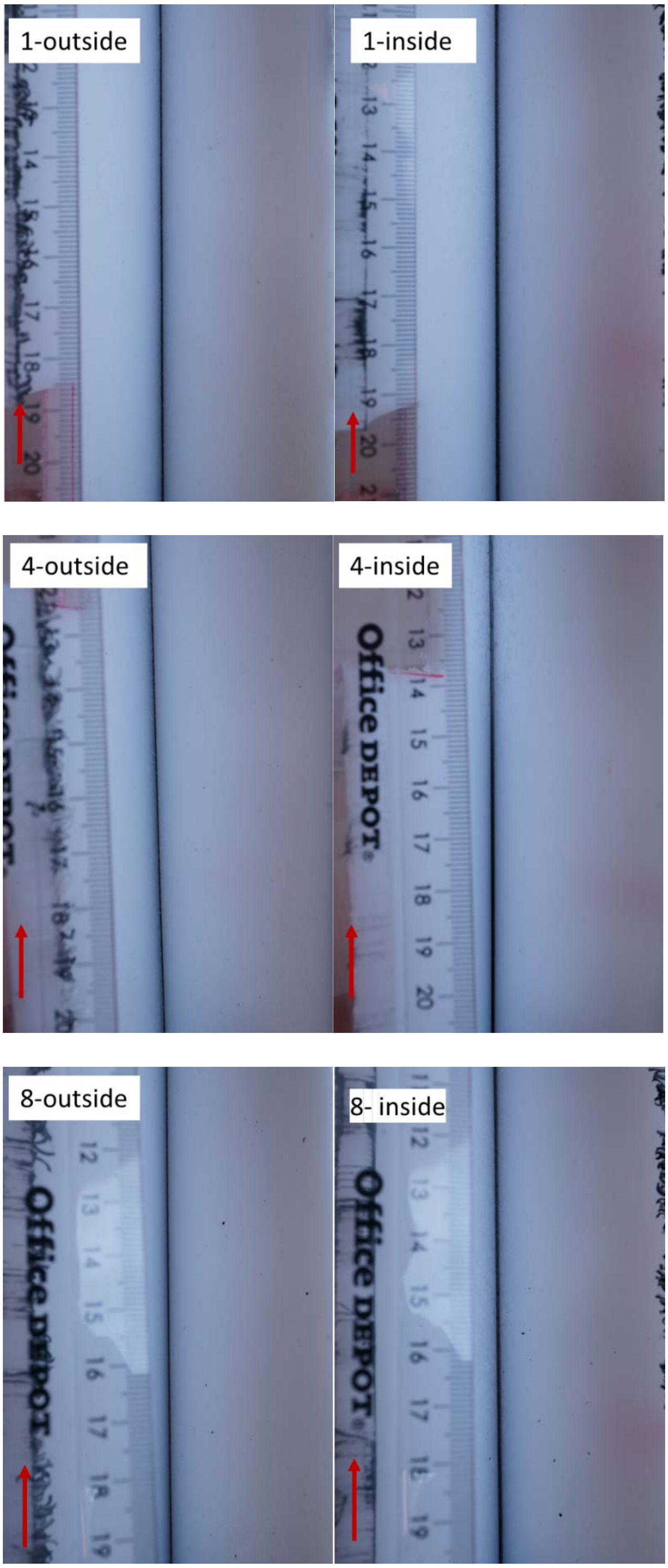


Figure 35: Optical images of individual bend segments 1, 4, and 8 taken from vertical direction. Left: outside half pipe; Right: inside half pipe

ii. Roughness and roughness distribution of the inner wall of eroded pipe segments

Virgin pipe

The WLI image of virgin pipe (Figure 36) shows a bit wavey appearance, with alternating blue, green and red regions. The green color in the WLI image indicates the zero height of the surface, blue and red present the dent and bulging on the surface respectively.

The roughness of the inner wall was determined by the statistics of the detecting points in the investigated specimens. The dashed line in Figure 37 indicates the height distribution of the inner wall of virgin pipe. It is clear that the inner wall of new pipe is not 100% even, with 3 height peaks corresponding to the stripes along the pipeline induced by the extrusion process.

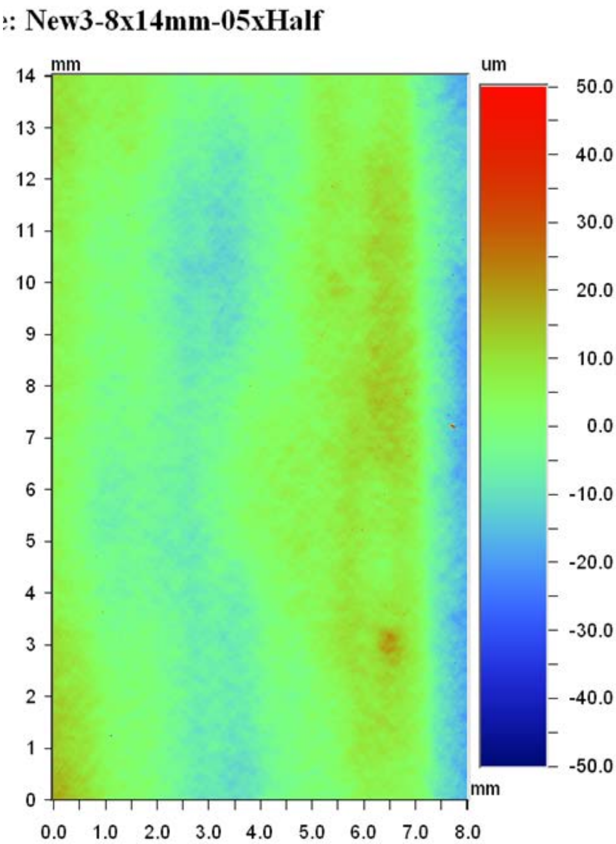


Figure 36: WLI image of inner wall of virgin pipe

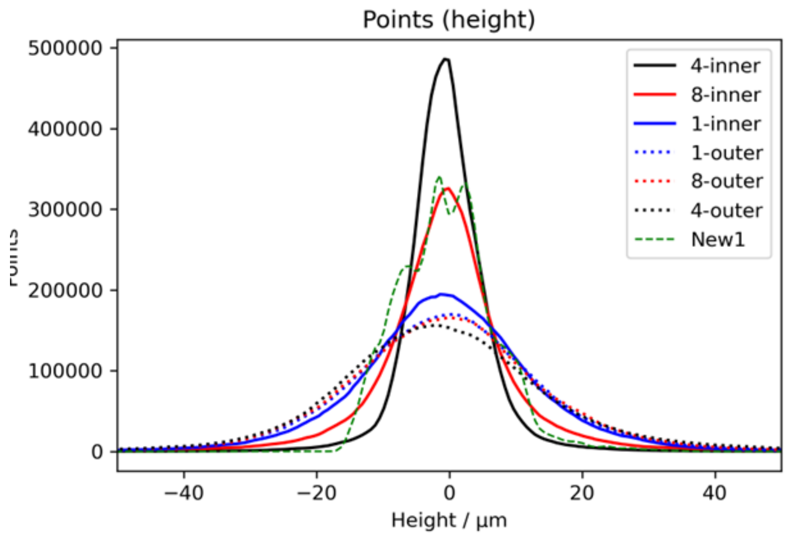


Figure 37: Height distribution of individual pipe segments

<u>Eroded pipe</u>

The WLI images of the inside and outside parts of segments 1, 4 and 8 are presented in Figure 38. The inside half and outside half of the Segment 1 show quite similar pattern with stretched scratches along the flowing direction of the feed pellets. The erosion patterns for two parts of segment 4 are significantly different, with deeper scartches condensely distributed in the examined specimen from outside half pipe, wheas as the inside half has sparasely shallow scratches. The outside half of segment 8 has less erosion compared to that of segment 4, but the inside half is more remarkably eroded. The WLI results is in good consistence with our observations in Section 5.3.1.

The above results indicate that all segments have a matt surface, suggesting the erosion of the inner wall by the conveying of feed pellets. The roughness and roughness distribution of the inner wall of the pipe segments indicate the degree and distribution of the erosion. Segment 1 has broad roughness distributions on both inside and outside half as can be seen from the roughness distribution in Figure 6, and the distribution curves are close to each other, indicating the homogeneous surface erosion. Whereas for segment 4, the outside half has a sharp and narrow distribution and the inside half has broad distribution, suggesting the outside half is deeply eroded whereas the inside half is least eroded among all the tested samples. Roughness distribution of segment 8 is between 4 and 1, with more erosion on the outside than the inside half, but the difference is not as significant as that in segment 4.

The morphological investigation of the eroded pipe indicates that the erosion of the pipe is varying upon the position of the pipe in the convey system, as different positioned pipe received varies impacts from feed pellets, and at the elbow of the bend, more pellets are accumulated, leading to severe erosion.

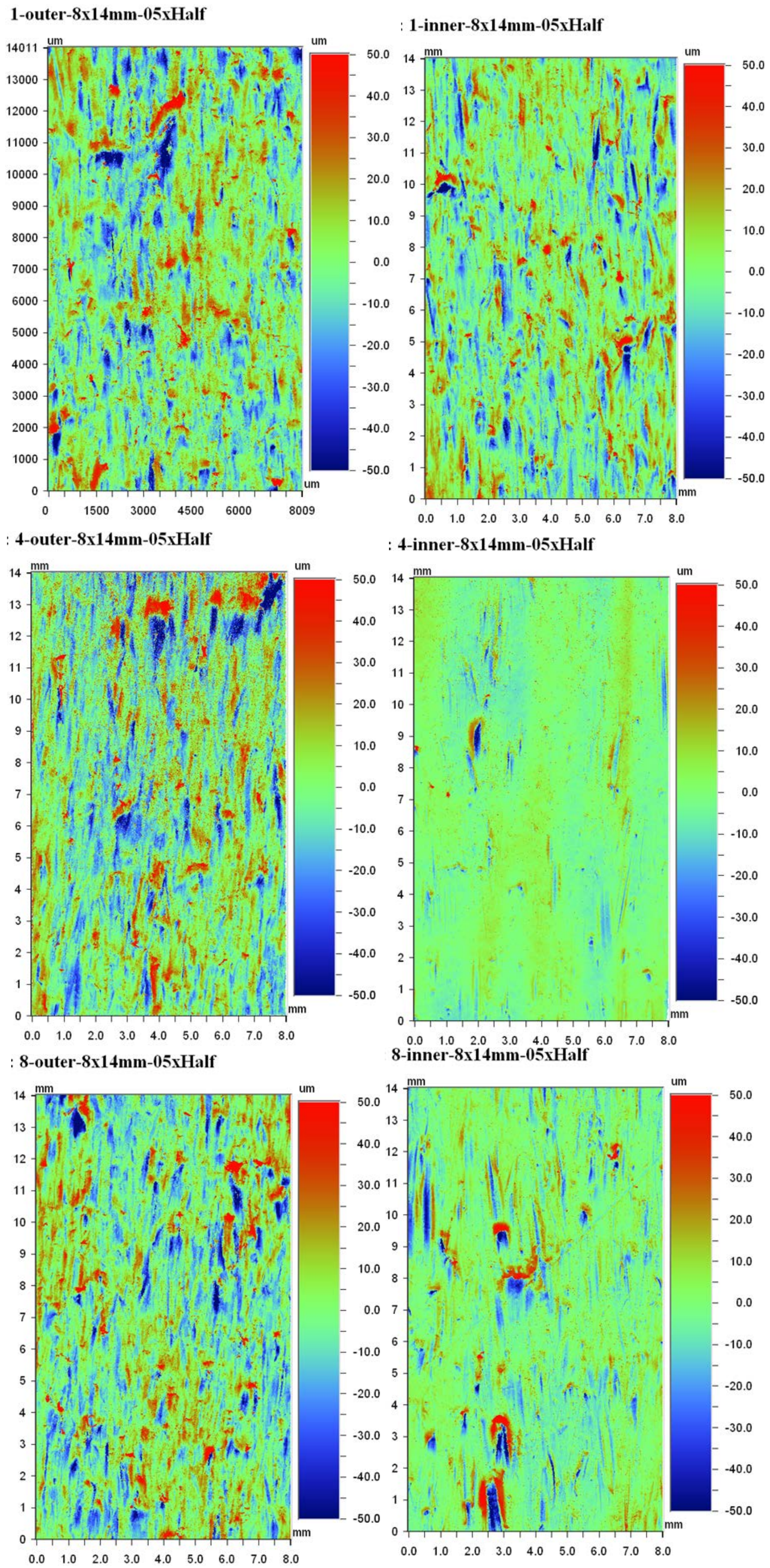


Figure 38: WLI images of inner wall of segments 1, 4 and 8. Left column: outside half pipe; right column: inside half pipe.

6) WP4- Implementation in fish farms and recommendations

a. Objectives

WP4 of MICRORED is aimed to develop a calculation program based on the results of the pilot-scale pneumatic conveying tests (WP1) on the scaling-up technique published elsewhere [8]. The calculation program allows prediction of the air conveying velocity and pressure profile along the pipeline with given input parameters for a given fish production facility (i.e., required transport capacity, available pressure and air volume flow rate, pipeline configuration, diameter and length). The simulation software will act as a tool for the fish farming facilities to optimize the operating conditions while keeping the air conveying velocity within acceptable limits, i.e., below the maximum threshold to avoid excessive erosion and pellet degradation and above the minimum limit to avoid pipe blockage.

The original plan included a part of testing and validating the results of the calculation program for a selected farming site, however, due to the challenges of finding a suitable site within the available time frame, the calculation program was demonstrated to the reference group discussing details of its applicability and relevant limitations.

b. Materials and methods

The test procedure with feed pellet's specifications and test rig's features are explained in detail under the section 3). As indicated in section 3)c.i, the test results of 'Pressure Tests' were used to make the calculation program.

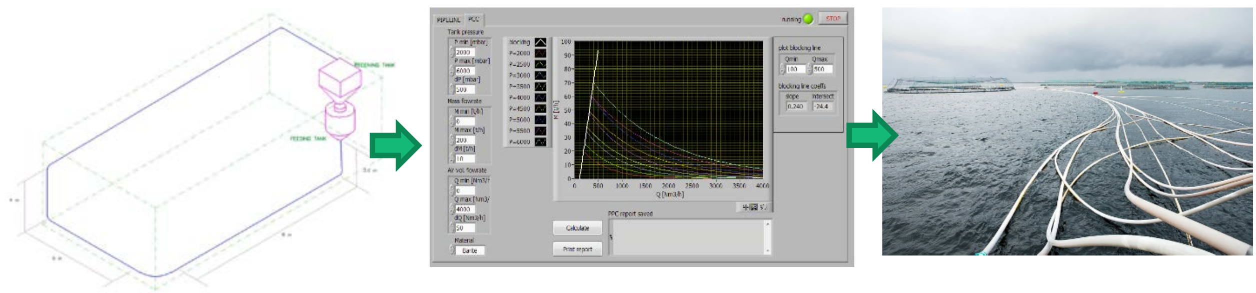


Figure 39: Schematic showing the development of the calculation program and its use in the fish farming site

c. Features of the calculation program

The program calculates pressure drops and conveying velocities in a feed pellet pneumatic conveying pipeline from the storage silo to the fish cage, according to user assigned values of the conveying parameters: supply pressure (**P**), air volume flowrate (**Q**) and transport rate (**M**). The program can further optimise the operation of a pneumatic conveying system through a systematic search for optimal conveying parameters.

Overview

The program has two modes of operation: **PLINE** and **PCC**, selected by their respective tabs at the top of the user interface. PLINE must be used to specify the pneumatic conveying line configuration and to investigate the conveying rig performance under a given set of conveying parameters. PCC is used to observe pneumatic conveying characteristic curves for the given pellet type and conveying line. These characteristic curves can be used for optimizing the conveying parameters P, Q, and M for the given conveying line and feed pellet type.

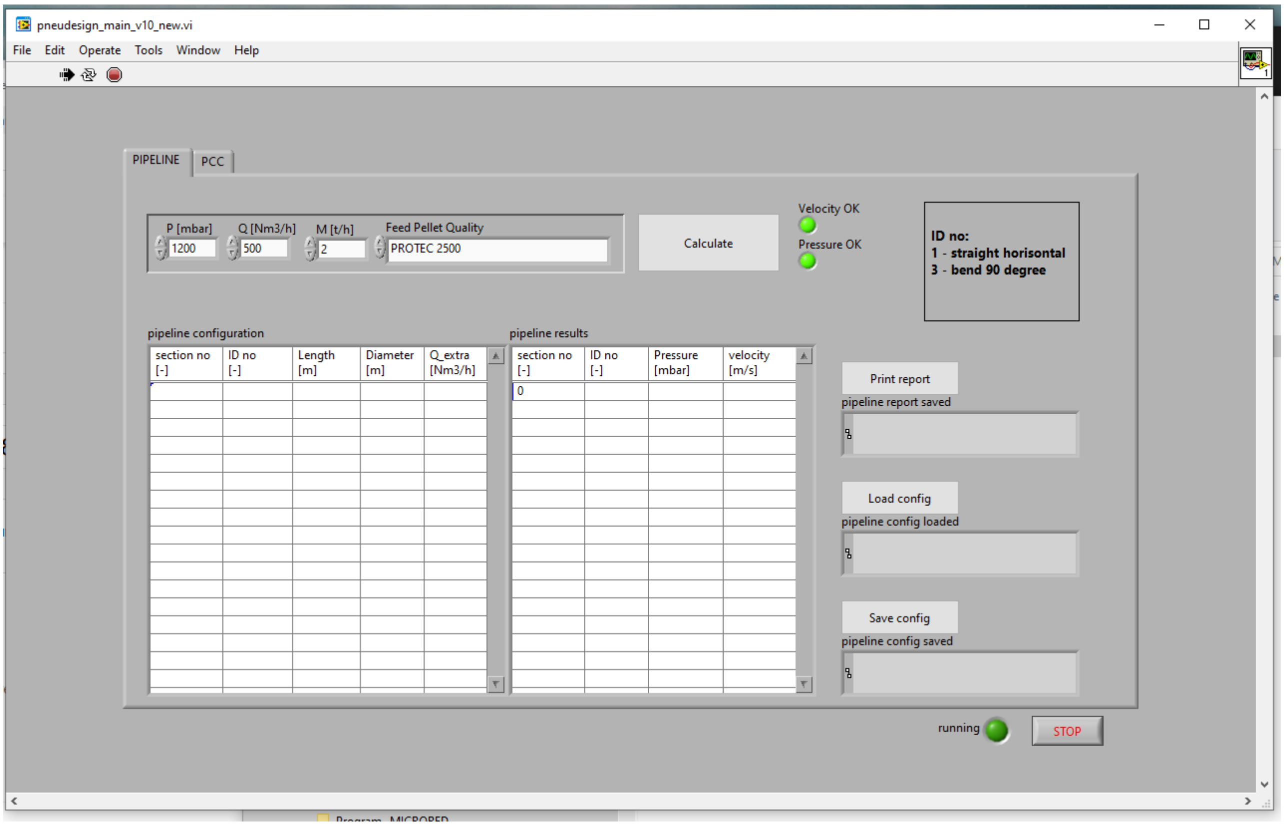


Figure 40: Start screen of the program

PLINE: Specify configuration of the conveying pipeline

The conveying pipeline is defined in the table “pipeline configuration”. A pipeline is specified by a number of sections, assumed to be connected in series and with no branching. Each section is further specified by:

Section no [-] : Number of the section, defining the geometry of the pipeline. First section is “1” and the final section has the highest number.

ID no [-] : type of section, denoted by a number: Straight horizontal (1) and 90 deg bend (2)

Length [m]: Length of the section. Note that calculated values of bends and valves are independent of length.

Diameter [m]: Diameter of the pipe section.

PLINE: Load pipeline configuration from Excel file

Left-click the “Load Config” button and select an appropriate Excel file describing the pipeline configuration.

A sample configuration file “pline_config_example.xls” is included with this installation. When the file has been successfully loaded into the "pipeline configuration" table and the "conveying parameters" control, the path of the loaded Excel file is displayed in the "pipeline configuration loaded" indicator box.

PLINE: Save pipeline configuration to Excel file

Left-click the "Save Config" button and select an appropriate path for the file to be saved. When the file has been successfully saved, the path of the saved Excel file is displayed in the "pipeline configuration saved" indicator box. Configuration of a complex pipeline may easily be continued using this Excel file, and subsequently loaded into the program with the "load config" feature.

PLINE: Specify conveying parameters

Simply enter the desired values for the supply pressure ***P*** in mbar, air volume flowrate ***Q*** in Nm3/h and mass flowrate ***M*** in t/h. Select material (barite, cement or tuned light cement) from the drop-down menu box.

PLINE: Calculate pressure drop and conveying velocity

Calculate the pressure drop and conveying velocity of the pipeline by left-clicking the "Calculate" button.

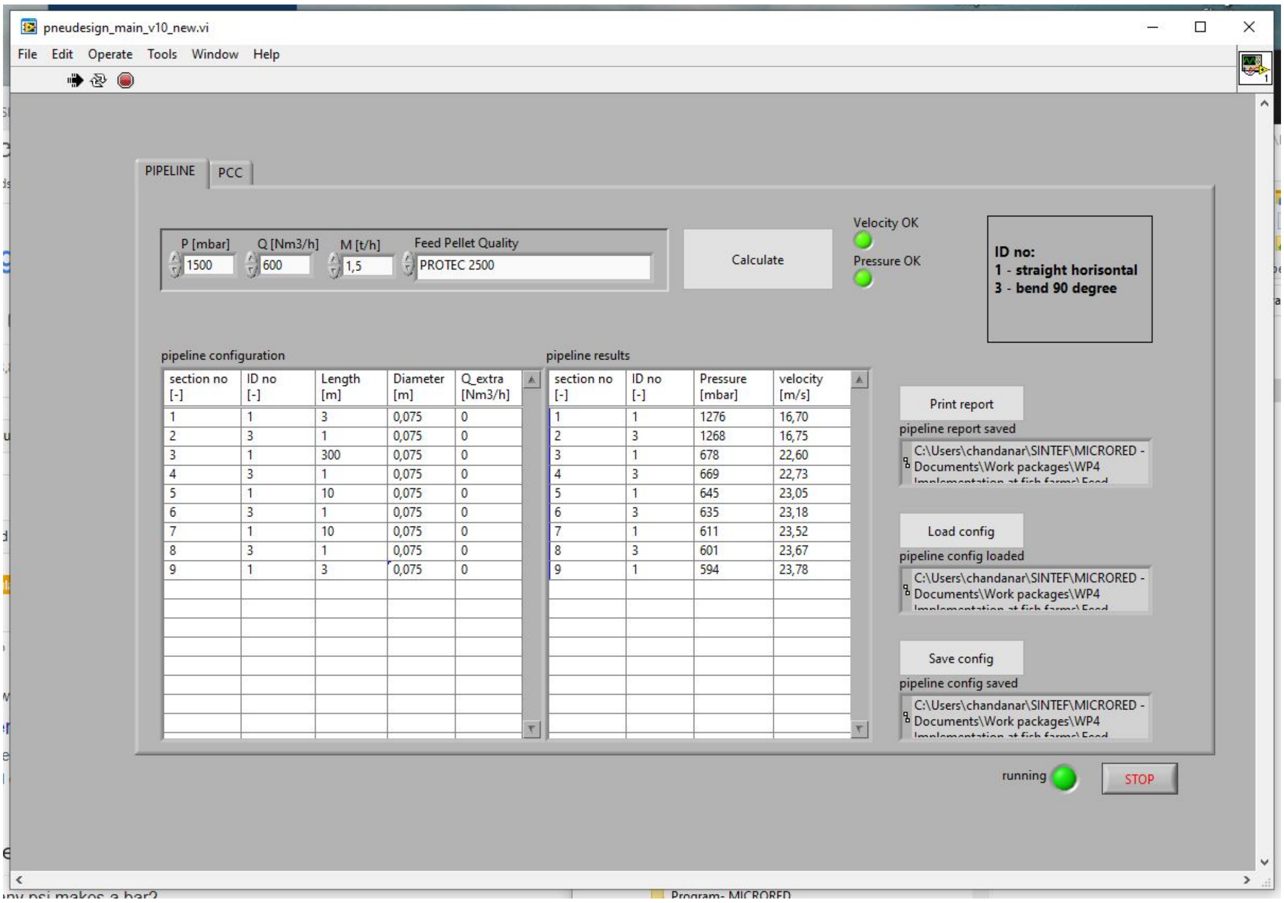


Figure 41: Screenshot of the program with filled information

PCC: Pneumatic Conveying Characteristic

The Pneumatic Conveying Characteristic plot shows the maximum permissible Transport rate ***M*** for the pipeline specified in PLINE as a function of air volume flowrate ***Q*** and supply pressure ***P***.

The program calculates the maximum ***M*** that satisfies the criterion of minimum outlet pressure drop and minimum conveying velocity in the range {***Mmin***, ***Mmax***} using steps of ***dM***. The conditions are calculated for supply pressure and air volume flowrate in the ranges {***Pmin, Pmax***} using steps of ***dP*** and {***Qmin, Qmax***} using steps of ***dQ***.

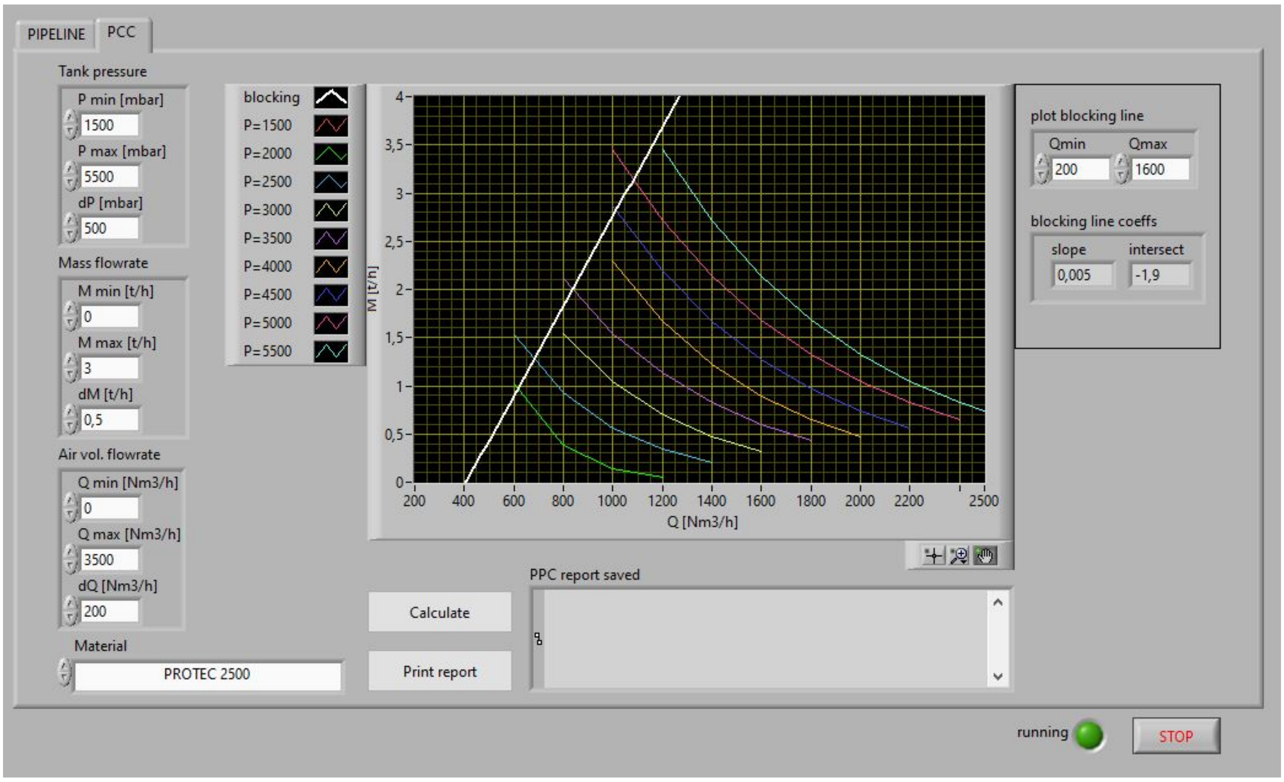


Figure 42: Screenshot of the program showing the pneumatic conveying characteristics (PCC) curves and relavant blocking line

PCC: Blocking Line

The blocking line (white trace in graph) denotes the maximum mass flowrate at a minimum air volume flowrate and a given blow tank pressure for the given pipeline configuration. This can be used to observe the conveying boundaries and to select optimized conveying parameters for a given pipeline configuration.

The coefficients of the blocking line are calculated for each click on the Calculate button. The blocking line is plotted for Q values given in the “plot blocking line: Qmin, Qmax” control. Re-calculating the PCC is *not* necessary for updating the plot of the blocking line.

d. Potential use and limitation of the calculation program

The calculation program developed using the experimental test results has many interesting usages in the full-scale feed pellet transport systems.

1 The program can be used in pellet transfer system design & optimization
2 It can be coupled with control systems to make sure the optimized operations
3 It can be coupled with 'standard' simulations (CFD, CPFD, +++) through UDF (user defined functions)
4 The approach can be used as a part of 'Digital Twin' of the transfer system connecting with available sensors

The program is based on the scale-up technique [9]; thus, it has some limitations too.

A. Only the tested quality of pellet can be simulated
B. Only the tested pipe components can be simulated
C. To include new feed quality and/or new pipe component, those should be tested in the pilot test rig and acquired necessary results

7) Main findings and conclusions

The results of the pneumatic conveying tests carried out by SINTEF Tel-Tek in a pilot-scale pneumatic conveying test rig with a commercial fish feed and a standard PE pipe show that in the range of conveying air velocities from 15 to 24 m/s the transport rate of pellets varies from 1.5 to 2.2 t/h. The data obtained from the tests were used to develop a calculation programme, and the report describes the potential of using it to optimize air-based fish pellet feeding systems at fish farms to minimise microplastic generations.

The plastic fragments characterization exercise performed by NORCE and SINTEF Ocean on samples obtained from the pneumatic convey system pointed out the formation of microns sized particles. Observed levels were dependant on where samples were collected in the conveying system. The samples distinct fragment size distribution pattern identifies a potential role of the fats layer coating the inner section of the feeding hose during standard fish feed supply. Such sticky and viscous material may trap plastic fragment and act in some cases as an impact adsorber increasing substantially the energy amount to induce the hoses abrasion. Based on the observed results there is no plastic fragments below 10 µm (nanometric size fraction) present in the observed samples. However, it is important to note that there are limitations with this approach. The primary one is linked to the lack of reference chemicals and the sensitivity study of the presented approach. Furthermore, the sample preparation procedure for both the enzymatic digest and extraction may have caused degradation and evaporative losses, respectively, of the target compounds.

The morphological investigation of the eroded pipe indicates that the erosion of the pipe is varying upon the position of the pipe in the convey system, as different positioned pipe received varies impacts from feed pellets., and at the elbow of the bend, more pellets are accumulated, leading to severe erosion. The content of fats coating the inner part of the feeding hoses play a role in the size distribution of the generated plastic fragments during the convey distribution simulation.

8) Main Findings (in bullet points)

- The commercial fish feed pellets can be conveyed with lower velocities than the industrial practices adopted in farming sites currently that will lead to minimise microplastic generation and/or pipeline erosion. The tested quality of pellet (Protec) could be transported in a standard PE pipeline within a range of conveying air velocities from 15 to 24 m/s acquiring a range of transport capacities from 1.5 to 2.2 t/h.
- The calculation programme developed in the project can be used to optimize air-based fish pellet feeding systems at fish farms to minimise microplastic generations, pipe erosion, ensuring pellets' integrity.
- The calculation program can be used in pellet transfer system design and optimization, and it can also be considered as a part of 'Digital Twin' of the transfer system. This can further be used with possible control systems, connecting with available sensors to make sure the optimized, trouble-free operations.
- The plastic fragments characterization exercise on samples obtained from the pneumatic convey system pointed out the formation of microns sized particles. Based on the observed results there is no plastic fragments below 10 µm (nanometric size fraction) present in the observed samples.
- The morphological investigation of the eroded pipe indicates that the erosion of the pipe is varying upon the position of the pipe in the convey system, depending on impact patterns of the pellets. The results highlight the potential of process optimization and pipe material developments to minimize microplastic generation and pipe erosion through further experimental investigations and/or simulation studies

9) Main Findings (in Norwegian)

- De kommersielle fiskefôrpelletsene kan transporteres med lavere hastigheter enn den industrielle praksisen som brukes på oppdrettsanlegg i dag. Det vil føre til mindre mikroplastdannelse og/eller fôringsrørerosjon. Den testede kvaliteten på pellet (Protec) kan transporteres i en standard PE-rørledning med transportlufthastighet fra 15 til 24 m/s og vil da oppnå en transportkapasitet fra 1,5 til 2,2 t/t.
- Beregningsprogrammet utviklet i prosjektet kan brukes til å optimalisere luftbaserte fôringssystemer for fiskepellets ved oppdrettsanlegg for å minimere mikroplastdannelse, rørerosjon, og å sikre pelletens integritet.
- Beregningsprogrammet kan brukes i utvikling og optimalisering av transportsystemer for pellets. Det kan også vurderes som en del av 'Digital Twin' av transportsystemet. Dette kan videre brukes med mulige kontrollsystemer, samt kobles til tilgjengelige sensorer for å sikre optimalisert og problemfri drift.
- Karakteriseringen av plastfragmenter fra det pneumatiske transportsystemet påpekte dannelsen av mikronstore partikler. Basert på de observerte resultatene er det ingen plastfragmenter under 10 µm (nanometrisk størrelsesfraksjon) i de granskete prøvene.
- Den morfologiske undersøkelsen av det eroderte fôringsrøret indikerer at erosjonen av røret varierer avhengig av rørets posisjon i transportsystemet, og av pelletens kollisjonsmønster. Resultatene fremhever potensialet ved prosessoptimalisering og utvikling av rørmaterialer for å minimere dannelse av mikroplast og rørerosjon gjennom ytterligere eksperimentelle undersøkelser og/eller simuleringsstudier.

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