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Waste Preparation Unit, concept and trade-off

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List of acronyms

CI : compartment I

MELiSSA: Micro-Ecological Life Support System Alternative

UAB: Universitat Autònoma de Barcelona

VFA: volatile fatty acids

BR: bioreactor

FU: Filtration unit

GL: Gas loop

FBD: Function block diagram

SFC: Sequential function chart

HMI: human interface

WPU: Waste Preparation Unit

EPAS: Eco Process Assistance n.v.

MPP: MELiSSA Pilot Plant

CEEAH: Comissió d'Ètica en l'Experimentació Animal i Humana

CSIC: Consejo Superior de Investigaciones Científicas

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1. INTRODUCTION

1.1. Objectives

The aim of this Technical Note is to describe all the tasks carried out for the definition and procurement of the Waste preparation Unit of compartment I of the MELiSSA Pilot Plant, according to the corresponding User requiremets already issued (RD2).

The work performed includes the definition of the technical specifications of the hardware, the trade-off among the different available technologies, the tests of some of them in order to select the best alternative, and the final purchase process.

1.2. Description of the tasks to be performed

The following are the main tasks performed within this definition and trade-off process:

- Evaluation of technologies available both technically and economically
- Performance tests on the selected technologies
- Trade-off
- Purchase of the unit

2. REFERENCE DOCUMENTS

Ref.	Title	Reference	Issue	Date
RD1	MELiSSA Pilot Plant: General Resources, Interfaces and Environment. MPP-TN-08-0011	MPP-TN-08-0011	0	01/04/08
RD2	WPU, User requirements and technical specifications	TN94.31	0	22/03/09
RD3	EWC Solid loop design (EPAS)	TN71.3	1	25/02/04

RD4	EWC Shipping procedure (EPAS)	Shipping procedure	4	30/07/07
RD5	MPP Hazard Analysis	MPP-TN-07-0001	3	30-03-08
RD6	EWC Design Report (EPAS)	Design Report	1	2006
RD7	Faeces donation donor´s procedure	MPP-OP-08-1001	0	24/01/09
RD8	Operation procedure for handling faeces samples	MPP-OP-08-1002	0	22/10/08
RD9	Procedimiento para la donación de heces. Información a los participantes y consentimiento informado	643H (CEEAH)	0	16/06/07
RD10	MPP Good Lab Practices Requirements	MPP-QA-07-0002	0	

3. RAW MATERIALS SUPPLY AND STORAGE

The raw materials defined for the use in the WPU are described in RD2 and RD3. They are herebelow recalled, including the supply needs for the MPP:

	Per week	Per month	Per year
Mass Faecal Material	700 g	3 Kg	36,4 Kg
Mass Toilet paper	126 g	0,540 Kg	6,550 Kg
Mass Wheat	380 g	1,628 Kg	19,760 Kg
Mass Lettuce	7560 g	32,4 Kg	393,1 Kg
Mass Beet	4760 g	20,4 Kg	247,5 Kg

Table 1. Raw materials needs for Compartment I

3.1. Faecal material

A program for faeces donation was established in the MPP, based on the following main requirements regarding the collection process:

- The needed amount of faeces will be in the range 80-120 g/day

- The faeces collection will be done inside a dedicated toilet near the MPP facilities, inside the Chemical Engineering Department.
- The toilet will be provided with W.C., shower and basin, and both toilet paper and disposable wet tissues. A specific waste bin will be available to be used if needed, and a cool box to keep the collected faeces samples.
- Airtight sterile sampling boxes specific for faeces collection and hygienic bags will be available inside the toilet.
- Faeces will be directly collected inside the boxes and these will be closed completely, placed inside the bags and stored inside the cool box.
- Collection of urine with the faeces should be avoided since human urine is not currently fed into the MELISSA loop for the pilot plant.
- Toilet paper will not be mixed with the faeces collected, but disposed separately.
- The boxes containing faeces will be maintained in the cool box during the day established for donation, and then transferred to the freezer inside the MPP by MPP personnel, where they will be maintained frozen at -18 ºC until use.
- Before freezing, the boxes will be weighted and identified indicating the collection date and the fresh weight.

All the issues regarding volunteer donation (including informed consent), confidentiality, health status of the donors, safety and traceability were as well addressed (RD7 and RD8), and the corresponding approval by the Ethics Committee for Experimentation (CEEAH) in UAB was obtained.

In order to obtain enough quantity of faeces samples for the continuous feeding of Compartment I , and taking into account that the donors are committed to supply a minimum of two faeces samples per week, it was defined a number of 4 as the minimum number of donors required, and it was decided to start the donation process two months ahead of the start-up of Compartment I tests, in order to store enough quantity of samples in advance to prevent any withdrawal of any of the donors from the donation for any reason.

Taking into account that the need of faecal material per week is 700 g, enough quantities of pre-weighted frozen samples of faeces will be selected in order to complete approximately this quantity in a weekly basis, and then the total quantity of feed to be prepared will be defined accordingly, so that the rest of the raw materials would be adapted to complete the recipe. In this way, the manipulation of defrosted faeces samples to be weighed is avoided: only frozen samples are used to be fed into the WPU tank.

3.2. Lettuce and beet

The fresh vegetable species selected for CI feeding were the following (RD3):

- *Lettuce Lactuca sativa L.*, variety Trocadero, including both edible and non-edible parts of the crop
- Red beet *Beta vulgaris*, variety Detroit, including the crop without leaves

In both cases, crops from biological culture are used, in order to guarantee a high quality of the crops and the absence of additives and other foreign substances to prevent as well the addition of any of these compounds to the MELiSSA loop. Both lettuce and red beet crops are provided by the company HORTEC (Mercabarna, Barcelona, www.hortec.org), a Catalan Cooperative Society of ecological agriculture, whose products are guaranteed by certified bodies on ecological agriculture. All partners of the Cooperative are certified by the Consell Català de la Producció Agrària Ecològica (CCPAE), the only entity authorized in Cataluña for this purpose.

In order to maintain as much as possible a regular feed composition to allow a stable culture conditions in Compartment I, it was necessary to guarantee a continuous supply of constant quality raw materials. Taking into account as well the fact that some concrete varieties of crops (especially regarding lettuce Trocadero) are harvested mainly during winter time, it was decided to store frozen the whole supply for one complete year. This was made in an external freezing warehouse: MERCATRADE (Mercabarna, Barcelona), that provides as well the periodic transport of the frozen crops to the MPP upon request.

3.3. Wheat straw

The dry vegetable species selected for CI feeding was Wheat *Triticum eastivum* (RD3), actually the straw, that is the non-edible part of the crop. In this case, the straw was provided to the MPP by local horse riding clubs or agricultural local markets (not certified).

3.4. Toilet paper

The white double-layer toilet paper "Renovagreen" was selected, from the European producer RENOVA (www.wellbeingworld.com), that provides a manufacturing process without the use of chemicals: 100% recycled paper; no dyes or inks; no fragrances; and whitened without chlorine. Renovagreen is certified with the European Eco-label.

4. REVIEW, STUDY AND EVALUATION OF THE WPU

4.1. General considerations

The Waste Preparation Unit is the first step in the feed preparation for Compartment I, The aim of the unit is to provide the adequate particle size of the raw materials (vegetables and faeces mainly) and to dilute them in water to obtain a homogeneus mix in order to be transferred to the feeding tank of the Compartment I.

As described in the User Requirements document (RD2), the selected parts of the higher plants need to be pre-treated, before introducing the material into the first compartment, up to a maximum particle size of 2 mm in the case fresh vegetables (lettuce and beet), and 0,2 mm in the case of dried ones (wheat straw). But to maintain stable raw material composition during the tests, a complete batch of lettuce and beet for approx. one year of use is frozen and stored before being treated. Regarding the manipulation of faeces, once collected they are frozen and their doses will be weighed first and then the required plant amounts will be prepared accordingly, in agreement with the recipe composition.

So the WPU receives as inputs: frozen faeces; frozen and pre-grinded vegetables; dried grinded straw and toilet paper in the ratio described in Table 1. The output shall be a suspension of all those raw materials in water with the specified composition (RD3, RD6) where the particle size will be lower than 0,2 mm for the straw and 2 mm for the rest of particles.

4.2. EPAS WPU (available equipment)

The description of the design and configuration of the WPU used in EPAS, and delivered to the MELiSSA Pilot Plant as part of Compartment I, is shown in RD4. It contained the following sections:

- Input vessel
- Kitchen grinder
- Recycle tank
- Submerged grinder pump
- Recirculation pipe with proportional valve
- Outlet pipe

The principle of operation of this WPU was as follows:

All raw materials (fresh vegetables, faeces, pre-milled straw and paper) were poured in the input vessel, where by the help of water pressure were transferred to the kitchen grinder. Once the mix had passed this grinder, it arrived to the recycle tank, where it was further processed by the grinder pump in order to reduce more the particle size. Finally, the mixture was recirculated through a proportional valve into the input tank again.
The system contained level switches in both tanks (high level switches in the recycle and input tanks, and low level switch in the recycle tank) to prevent dry operation of the pump and the water overflow, respectively, controlling the pump and the automatic water inlet

valves by means of a small PLC. Manual valves were used both for the outlet of both tanks and for the recirculation.

The materials used in EPAS WPU are listed in Table 1. Both the quality and the design are considered not adequate for the requirements regarding both quality and safety/hygiene defined by the MELiSSA Pilot Plant (RD5 and RD10). The following main points were identified as potential risks in this sense:

- Piping, valves and connections in PVC, not sanitary
- Tanks material not sanitary: Stainless Steel 304 (low quality), not polished internally
- Submerged pump: difficult to clean (not removable); shell and cutting parts not in stainless steel
- Kitchen Grinder: cutting parts not in stainless steel
- Not steam sterilisable

Description	#	Brand	Article	Supplier
Electrical Cabinet 500mm x 500mm x 210mm	1	Rittal	1050600	Rittal
Level Switch	3	E+H	Liquiphant T FTL20: FTL20-0 0 10	E+H
Stainless steel 304 construction (frame with tank, supports and piping)	1	SE Industries	-	SE Industries
Grinder pump	1	DAB	RT AD 2015-2M S/A	DAB
			B2085 Motor ginder ISE 0,75pk Single Phase 240v 50/60hz	
Grinder	1	SISSONS		EFAS
Electrically actuated valve (open/closed)	1	Bürkert	Model 6013: 134 259 B (p 5)	Bürkert
Electrically actuated valve (open/closed)	1	Bürkert	Model 6013: 134 259 B (p 5)	Bürkert
Manuel ball valve with solvent cement sockets 40 mm	1	+GF+	163 546 015	+GF+
Manuel ball valve with solvent cement sockets 20 mm	1	+GF+	164 546 012	+GF+
Manuel ball valve with solvent cement sockets 50 mm	2	+GF+	164 546 016	+GF+
PVC Stop 3/4"f	1	+GF+	721 961 907	+GF+
PVC Adaptor fitting elbow 90° 40mm to 1 1/4" female thread	1	+GF+	721 100 209	+GF+
PVC Socket 40mm to 1 1/4" female thread	3	+GF+	721 910 209	+GF+
PVC adaptor 3/8" to m-16	2	+GF+	721 910 705	+GF+
PVC union for solvent cement jointing 40 mm	4	+GF+	721 510 134	+GF+
PVC Elbow 40 mm	2	+GF+	721 100 109	+GF+
PVC Elbow 45° - 40 mm	2	+GF+	721 150 109	+GF+
PVC bend 90° - 40mm	1	+GF+	721 000 109	+GF+
PVC short reducing bush 40mm to 25mm	2	+GF+	721 900 347	+GF+
O-ring FPM, d=20 mm	5	+GF+	749 410 006	+GF+
Nozzle	2	PNR	UAA 1228 B3 ZB	PNR
PLC	1	Schneider Electric	Zelio 240VAC, 12 IO, SR1A201FU: 3783029	FarnellInOne
Circuit Breaker C60H	1	Merlin Gerin	24988	Cebeo
Realais 20A 230VAC	2	CTX	666131	Cebeo

Table 2. Materials used in the WPU delivered by EPAS

The WPU unit received from EPAS was partially dismantled and its status was visually checked (Fig.1) . Corrosion both in the submerged pump and in the kitchen grinder rotor was clearly appreciated, corroborating the considerations on the materials quality above described

Fig.1. Status of the WPU delivered by EPAS: a) submersible pump; b) Kitchen grinder rotor

Regarding the particle size, in RD6 it is stated the requirement of 2 mm for the mixture obtained in the WPU. Nevertheless, there is not recent evidence about the size distribution of particles after processing the mixture of raw materials by means of this equipment.

The conclusion of this study was that a new WPU should be designed and installed in the MPP, coherent both with the User requirements already defined and with the requirements regarding both quality and safety/hygiene defined in the MELiSSA Pilot Plant.

4.3. Trade-off of Milling techniques and systems

In Table 2 a summary of the techniques and systems evaluated for the objectives of the WPU is shown. The following criteria were taken into account for the selection:

- Type of equipment proposed: mainly technology, size and power consumption
- Prerequisites: previous milling or preparation of the raw materials
- Particle size obtained

- Price
- Availability of tests

COMPANY	EQUIPMENT PROPOSED	PREREQUISITES	PART. SIZE OBTAINED	PRICE	TESTS
BACHILLER (SPAIN)	VEGETABLES: BLADE CRUSHER + DRYER STRAW: COFFEE MILL + SIEVING	FREEZING DRYING	< 1 mm 250 μm	DRYER: 50500 € VAC. PUMP: 22300 € MILL: 42800 €	TESTS NOT AVAILABLE
FRISTAM (GERMANY)	SHEAR PUMP	CUTTING TO ≈ 1 cm	PARTICLE SIZE >2 mm (ESPECIALLY STRAW)	≈ 6000 €	TESTS AVAILABLE IN MPP: - KIND OF ROTOR - TIME - SPEED
IKA (GERMANY)	A) KNEADING MACHINE B) ULTRATURRAX	CUTTING TO FEW mm	2 mm < 1 mm	25000 € 18000-20000 €	TESTS AVAILABLE IN MPP OR IKA LAB
NETZSCH (GERMANY)	MILLS (DIFFERENT TYPES)	CUTTING TO 100 μm	nm		
MOREHOUSECOWLES (US)	MIXER-DISSOLVER				TESTS AVAILABLE (SIMILAR EQUIPMENT)
VAK KIMSA (SPAIN)	IN-LINE MIXER VISCOVAK			≈ 23000 €	TESTS AVAILABLE
RETSCH (GERMANY)	VEGETABLES: GRINDER STRAW: CENTRIFUGUE MILL	CUTTING TO 8 mm	PART. SIZE NOT GUARANTEED SEQUENTIAL MILLING AND SIEVING (8→2→0,2 mm)	MILL:6100 € SIEVES: 400 €	ALREADY IN USE BY EPAS TESTS AVAILABLE
FRITSCH (GERMANY)	STRAW: CUTTING MILL	CUTTING TO 8-10 mm	Min. 0,25 mm	MILL: 7000 € SIEVES: 300 €	AVAILABLE IN CREAF (UAB)
FOSS (SWEDEN)	STRAW: SAMPLE CYCLON MILL		Min. 0,5 mm		AVAILABLE IN UAB

Table 3. WPU preliminary trade-off

4.3.1. Vegetable milling techniques and systems

Regarding the type of equipment, basically four types of them were proposed: blade crusher, shear pump, kneading machine, mills and grinders.

a) BACHILLER (Barcelona, Spain)

Profile of the company: this company works mainly on major projects on machinery and chemical engineering, but deal also with R&D projects, mainly for chemical and food industry (www.bachiller.com). For some applications, they cooperate with concrete Food R&D Centres, in order to carry out some trials for new equipment. That was one of the reasons to contact them.

- <u>General considerations</u>: having into account the kind of materials to grind, the unknown component is faeces (vegetables behaviour is well-known), and they are supposed to enhance the difficulty of grinding at small sizes. So a particle size of less than around 2 mm (perhaps 1,5-1,8 mm) could not be guaranteed, even using frozen vegetables, frozen faeces, and straw in a separate milling. According to this company, there were applications using frozen vegetables where 1 mm particle size is achieved (frozen pepper or onion), but not including faeces.

- Machinery recommended: they proposed to use a blade crusher, its working principle being based on a rotor equipped with blades, revolving at a low speed through the gaps in the grid type stator. A screen plate would be incorporated to achieve the desired particle size. Due to their strength, SB crushers can handle products as hard as solid resins, while they may also be used for other materials such as vegetable products. The design allows the blade crushers to be incorporated easily either in the feeding or in the discharge section of any processing machine, as well as being used as a pre-grinding unit prior to micronization .
To guarantee freezing during the process, a liquid nitrogen inlet can be installed wetting the materials at the same time they are milled.
It would be a 3 Kw machine, around 60-70 cm width, with a hopper and a screen plate, completely manufactured in stainless steel, easy to open for cleaning. Downstream the machine they could provide any system for mixing and wetting in connexion with CI, as close as possible.

This machine is being used for similar applications in frozen vegetables (carrot and onion milling) and seaweed milling.

- Recommendations to reduce the particle size: in the case of vegetables (without faeces), they considered possible to further reduce the particle size even up to 0,25 mm, but to achieve this, vegetables need to be first dried (<0,5% water). That means to install a dryer before the mill, being the investment considerably higher. In the case of vegetables+faeces, they did not guarantee such a low size even drying the components, nevertheless considering achievable a size around 1 mm.

The positive aspect of this equipment was the low particle size that could eventually be obtained, and the power required for the equipment is not so high. However, to guarantee this size to be around 1 mm a previous step of drying should be installed, and then both the power consumption and the investment should be much higher. Beside this, the investment of just the milling equipment was more than 40 K€, and performance tests were not possible.

b) FRISTAM IBERICA (Girona, Spain)

This company proposed a Shear pump: the mixing method is based on the proven centrifugal pumps of the Fristam FP series. Instead of the impeller, a rotor/stator system operating at tip speeds of up to 38 m/s, draws inhomogeneous media through shearing clearances of just 0.3 mm. Extremely high flow rates generated in the rotor/stator system and a high shear rate of up to 125,000 s^{-1} enables high-performance blending of multiphase products. The result is inseparable emulsions and end products of high homogeneity.

Since applications vary in type and complexity, customised solutions range from small single units to large-scale inline installations.

Compared with conventional dissolving processes in large tanks or boilers, using the Fristam Shear pump can cut the processing time by up to 90 %. The Shear pump disintegrates agglomerates, lumps etc., thanks to its high shearing energy, and delivers absolutely constant, reproducible results. This pump is also highly suitable for handling varying batch sizes. The forced flow of products in the shear pump ensures a constant high quality standard.

Depending on the application, the use of less raw materials can be expected because of the more effective breakdown of constituents.

Cleaning and maintenance: the Shear pump is fully CIP adapted. Due to the similarity in construction with the centrifugal pumps, the user can usually take care of cleaning and maintenance by himself.

The investment for the FRISTAM shear pump was more affordable; beside this, the fact that performance tests are possible even in the MPP, was another advantage, as well as the CIP and SIP possibilities. However, the need of previous cutting, the power consumption and the level of noise of this pump were some of the identified disadvantages.

c) FAUST AND KAMANN – IKA (Barcelona, Spain)

This company proposed a horizontal kneading machine for the grinding of vegetables. A very large viscosity range from – for a kneading machine – rather low-viscous glues (hot melts) up to extremely high viscous rubber mixtures can be processed with the IKA® horizontal kneading machines with specific blades. The use of these kneading blades patented by IKA results in a substantially improved product homogeneity and a saving of up to 30% of kneading time compared to the classical Z-kneaders. Vacuum-tight and double jacketed kneading bowls, high quality stainless steel for all parts in contact with the product and high-quality shaft seals with easy access for service and maintenance are part of their basic equipment.

The common applications of this technique are: glues (e.g. hot melts), rubber masses, plastic mixtures, ceramic masses, porcelain, colour mixtures (e.g. printing inks), carbon pastes, graphite mixtures. The equipment provides options like e.g. discharge screw, removable sidewall or removable kneading bowl.

After the discussion with this supplier, the main disadvantages considered were: the high investment needed, to be a batch processing (continuous would be extremely expensive) and the fact that no previous experience was demonstrated on the size reduction of vegetables, although it was possible to perform some tests in advance of the purchase even if involving some added cost.
As an alternative, the "Ultraturrax" option was proposed , a single stage rotor-stator-system for homogenizing, emulsifying and suspending tasks. An equipment similar to this the VAK KIMSA in-line mixer, herebelow described. In this case, an in-line application would mean a lower investment compared with the kneading machine.

d) NETZSCH ESPAÑA (Barcelona, Spain)

This company supplies all kind of mills, both for dry milling and wet milling. But in general the prerequisite for their equipment is already a particle size not higher than 100 μm in the inlet stream, then being able to reach a final particle size in the order of nm.

This was then considered out of our scope, because it would require an additional equipment even upstream of this one.

e) VAK-KIMSA (Barcelona, Spain)

This company is specialized in development and manufacturing of blending equipment. They build in-line mixers installed outside the reactors and used for blending processes, both to operate in batch or continuous, with the advantage of providing lump-free mixtures. The proposed equipment for our application is the VISCOVAK, machine manufactured to produce thickening solutions, creams and sauces. Their specific rotor and stator designs are confidential and the cost of the unit is quite high (around 23 K€). However, a test in their Pilot Plant was offered and is described in Section 5.7.

f) MOREHOUSE COWLES (US)

MorehouseCowles is a well known American company in milling technology. MorehouseCowles' line of single shaft dissolvers are designed for liquid to liquid or dry-to liquid mixing for a range of product viscosities. Direct drive mixers range from 5 to 125 horsepower. Fixed or variable speed mixers range from 1 to 300 horsepower. All are available with mechanical or electronic variable-speed drives (see Figure 2 for an standard industrial equipment).

Figure 2. Industrial standard Cowles equipment and "hi-shear" impeller.

Considering that a similar equipment was available in the Food Processing Pilot Plant in UAB, a test was performed and is described in Section 4.7.

4.3.2. Straw milling techniques and systems

g) BACHILLER (Barcelona, Spain)

They considered better to use an equipment like a coffee mill, dedicating a long time of milling, and a manual sieving after that, to be mixed with the other components. There was not a formal proposal from this company.

h) RETSCH

The rotor mill series includes preliminary size reduction sample dividers, ultra centrifugal mills, rotor beater mills and cross beater mills. Depending on the particular instrument they are suitable for the preliminary and fine size reduction of soft, fibrous and also hard materials. A final fineness of down to 40 µm can often be achieved in the first working step. The maximum feed size depends on the mill and ranges from 10 to 15 mm. Material which is larger than this must first undergo preliminary size reduction.

The Ultra Centrifugal Mill from RETSCH ensures higher performance with increased torque. The machine even reacts to temporary overloads with a continued throughput which ensures particularly efficient grinding. The extremely quick size reduction increases sample throughput in the laboratory and, in combination with the 2-step rotor-ring sieve system, is also extremely gentle on the material. Soft, elastic products such as plastics, which do not process well at room temperature, could be fed into the mill after embrittlement with liquid nitrogen or dry ice.

The RETSCH Ultra Centrifugal Mill is used for the rapid fine size reduction of soft to medium-hard and fibrous materials. Because of the efficient size reduction technique and the comprehensive range of accessories the mill ensures the gentle preparation of analytical samples in a very short time.
The suitability of the ultra centrifugal mill for universal use is shown by the following examples: chemicals, drugs, spices, coal, synthetic resins, plastics, pharmaceutical raw materials and finished products, fertilizers, nitrogen and protein determination in food and feedstuffs, environmental studies into plant constituents, preparation of bones, fossil fuels and secondary fuels, sample batch production of customized powder coatings, surfactant determination in washing powders. The ultra centrifugal mill is used in both quality control and R&D.

In the ultra centrifugal mill size reduction takes place by direct impact and shearing effects between the rotor and the fixed ring sieve. The feed material passes through the hopper (with splash-back protection) onto the rotor. Centrifugal acceleration throws it outward with great energy and it is pre-crushed on impact with the wedge-shaped rotor teeth moving at a high speed. It is then finely ground between the rotor and the ring sieve. This two-step grinding ensures particularly gentle but fast processing. The feed material only remains in the grinding chamber for a very short time, which means that the characteristic features of the sample to be determined are not altered. The ground sample is collected in the collecting cassette surrounding the grinding chamber or in the downstream cyclone or paper filter bag.

i) IZASA - FRITSCH (Barcelona, Spain)

FRITSCH proposed for the milling of the straw a cutting mill. Cutting mills are particularly well suited for comminution of soft to medium-hard, fibrous or tough materials (including plastics). They are frequently also used in the preparation of heterogeneous substance mixtures. In this type of mill, samples are comminuted through cutting and shearing. The fineness is determined by a sieve insert. In cutting mills, it should be possible to remove the grinding parts with minimal or no use of tools so that cleaning can be performed quickly and efficiently.

For control of the wearing and abrasion, hard metal tungsten carbide can also be used as the grinding tool material in place of various steels. The cutting mill combination and the power cutting mill are particularly well suited for coarse comminution (e.g. environmental work).

Applications: Plastics and textiles; agriculture and forestry; environmental wastes milling; analysis; construction materials; chemistry; foodstuffs.

There was a cutting mill supplied by this company already existing in UAB, in concrete in the CREAF (Centre de Recerca Agricola i Forestall), that could be used for comparative tests using MPP samples of straw (see tests carried out in Section 4.7).

j) FOSS-TECATOR (Barcelona, Spain)

This company manufactures sample lab mills like the Cyclotec, that is designed for rapid, uniform grinding of a wide variety of dry samples. The Cyclotec is based on the cyclone principle for universal grinding applications in the laboratory. The grinding is carried out by a high-speed action whereas the sample is rolled against the inner circumference of a durable grinding surface and passed out through a screen. The sample is fed on to a turbine wheel that spins at a very high speed, breaking the sample into pieces and hurling them out to the rim where they are abraded to a fine powder. The turbine also acts as a fan, so that particles of the requisite fineness are carried away with the air flow through the screen and separated in the cyclone.

The high grinding capacity reaches 4 g/second and the recovery of the sample is complete, meaning efficiency and accurate preparation for several analytical techniques eg. Kjeldahl, extraction etc.

The mill is made for a variety of dry samples such as feeds/grains whereas the sample is rolled against the inner circumference of a durable grinding surface and passed through a screen. No moisture losses and fine, uniform grist imply reliable and accurate analysis.

The grinding impeller stops working when the grinding chamber is opened, which assures a reliable usage of the mill. High volume airflow provides a self cleaning action, whereas whole series of samples can be ground without clean out in between which saves time and costs. Less maintenance reduces the running costs and contributes to an easy handling.

There was a Cyclotec supplied by TECATOR already in UAB, in concrete in the Department of Animal and Food Science (Veterinary Faculty), that could be used for comparative tests using MPP samples of straw (see section 4.7).

4.4. Preliminary selection

Vegetables (lettuce and beet):

Taking into account all the technologies and equipment above described, the investment required and the particle size foreseen to be obtained were the main parameters to consider in a first step of the selection.

High investment and the need of pre-freezing or drying, as well as the fact that realization of preliminary tests were not possible, were the main arguments to discard the alternative of blade crusher from BACHILLER.

In the case of IKA, a high investment was as well the reason to discard the kneading machine, considering as well that there was not previous experience with the application required for the MPP.

The equipment proposed by NETZSCH was in fact oriented to a further step of milling, requiring already a previous equipment for its purpose, so it was as well discarded.

So the alternative selected was the shear pump supplied by FRISTAM, considering the lower investment, availability of tests and SIP and CIP adaptability, so a testing phase was agreed with the supplier in order to define the final design of the equipment, to be used in-line.

Wheat straw:

For the wheat straw milling, on one side it was clear after the discussion with the different suppliers and the experience of EPAS, that a dry wheat milling was needed, then separated from the milling of lettuce and beet. Considering that some equipments were already available in UAB and tests were as well available for the RETSCH mill, the decision was to perform enough tests to assess the particle size distribution, taking into account as well the appropriate method of measurement, because in the case of straw, being a fibrous material, this is in fact a relevant point for a proper decision (see section 4.7).

4.5. Tests carried out with the FRISTAM shear pump

The first tests were carried out in the MPP with the FRISTAM shear pump Model FSPE 3532 (7,5 KW, up to 3000 rpm), together with a circulation tank and piping, and a regulating valve (Figure 3).

Figure 3. FRISTAM shear pump prototype and ancillary equipment for testing in the MPP.

Both the speed, appropriate flow, rotor shape and time of operation were tested in order to obtain a continuous grinding and to avoid blocking the rotor-stator system. Different samples were taken at different times of milling, that were further diluted, filtered and the particles measured (by means of a magnifying glass and an associated ruler).

The raw materials (lettuce, beet and wheat straw) were pre-grinded in a food industrial Cutter (Food Processing Pilot Plant, UAB).

First tests were performed with separate suspensions of lettuce, beet and straw. Later on, mixed suspensions of the three components in the concentrations foreseen for the WPU nominal operation conditions were used.

The main conclusions obtained were the following:

- The maximum particle size acceptable for feeding the pump was about 1 cm. Bigger particles blocked the pump or were trapped among the rotor-stator teeth. This was especially clear in the case of fibrous materials like the straw (Figure 4).

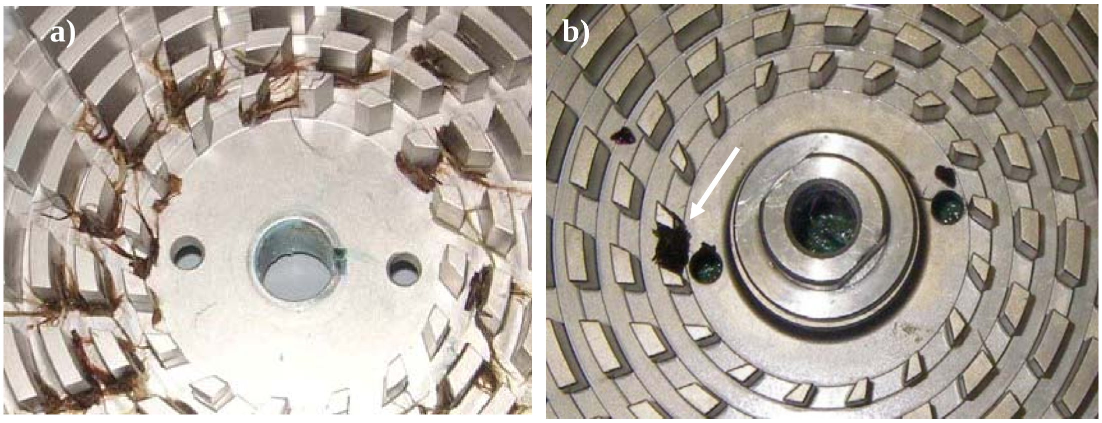
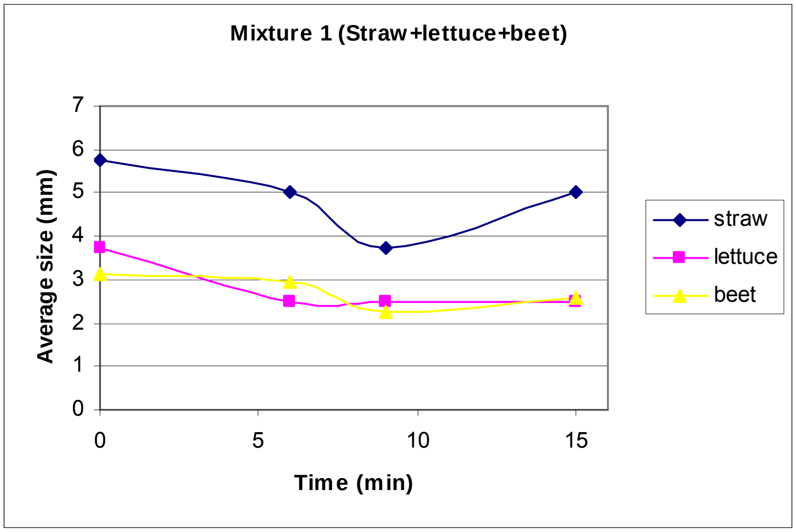


Figure 4. Retention of big particles in the shear pump rotor: a) straw particles; b) beet particles

- Given that pre-grinded vegetables are used (≤ 1 cm), it is not necessary to implement an additional pump for the feeding of the circuit, because the sucking capacity of the shear pump is enough for the operation. However, in order to avoid the foam formation, the recirculation pipe outlet should be preferably immersed in the medium of the recirculation tank.
- The effectiveness of the milling process to reduce the particle size was somehow relevant in the beginning of the operation (up to 5 min aprox.), being after that only slightly effective; in particular, the straw particles seemed to maintain its length due to their fibrous character and long shape (See Figure 5). In Figure 6, some of the samples collected are shown on paper filter for observation and measurement.
- Three kind of rotors were tested, different both in the number and dimensions of the teeth, and in the position of the same respect to the ones in the stator, so causing different shear effect. (See Figure 7): in rotors 1 and 2 the teeth size increases from the center to the periphery, helicoidally distributed (radially in the stator) ; in rotor 1 the number of groups of teeth coincides with the one in the stator, but in rotor 2 is different, to increase the shear effect; rotor 3 (standard) has equal size teeth spread all along the rotor.
- Two kind of stator were as well tested, one of which (Stator 2) is shown in Figure 8: as it can be observed, its teeth are drilled, in order to increase the cutting surface. The results obtained with the three rotor configuration and the two stator ones are shown in Table 4: the particle size obtained after 5 min of milling is similar in all the conditions, although the best result was with Rotor 1 and Stator 1. The differences are probably not significant taking into account the few number of measurements and the high dispersion of the results (std dev. >40%).



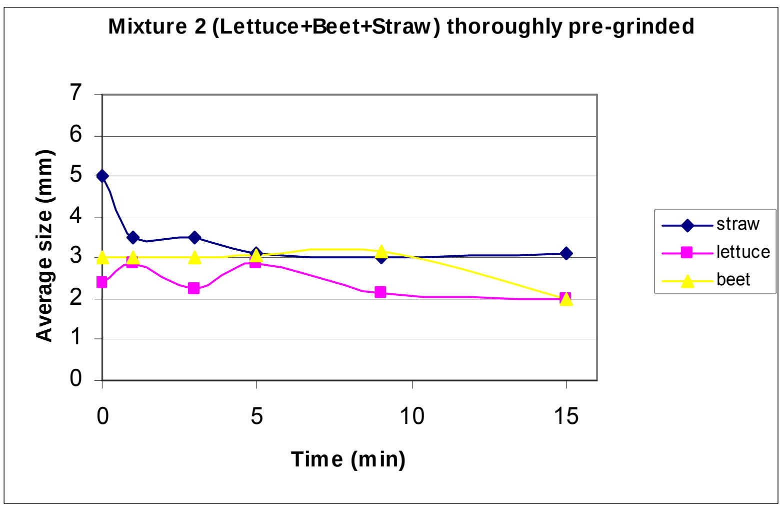


Figure 5. Reduction of particle size with the shear pump (first test)

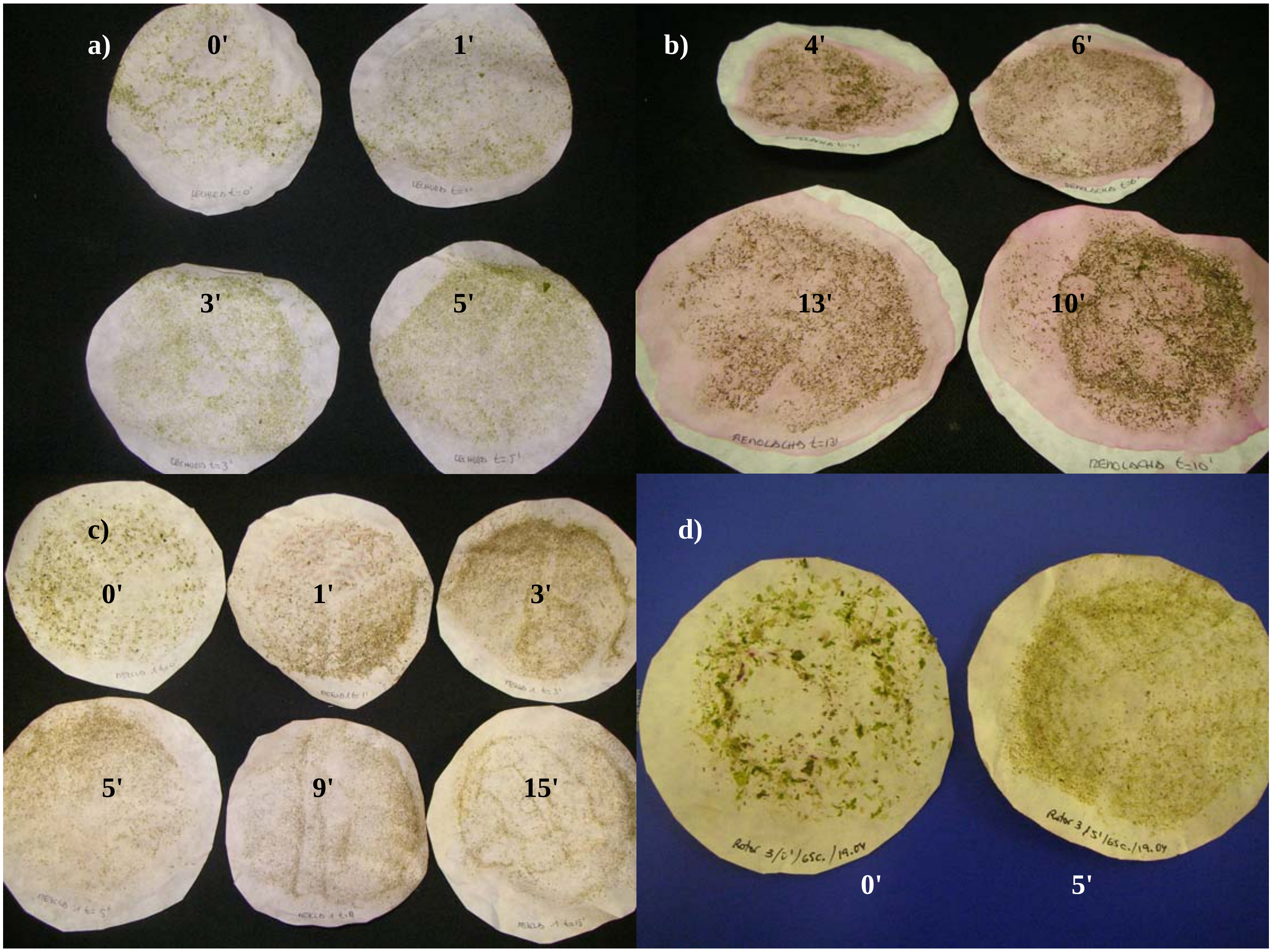


Figure 6. Different samples obtained during the milling process with the shear pump: a) lettuce milling at different times; b) beet milling at different times; c) milling of whole mixture at different times; d) Sample of the initial mixture (left) and the same mixture after 5 min of milling (right)

- The behaviour of fresh and frozen pre-grinded vegetables was quite similar in terms of efficiency of the shear pump, as can be observed in Table 5.
- Comparing the different times and speed of milling tested (Table 6), no significant differences were observed between 5 and 10 min of operation of the shear pump, and the increase in speed from 3000 rpm to 4000 rpm was as well not clearly effective.

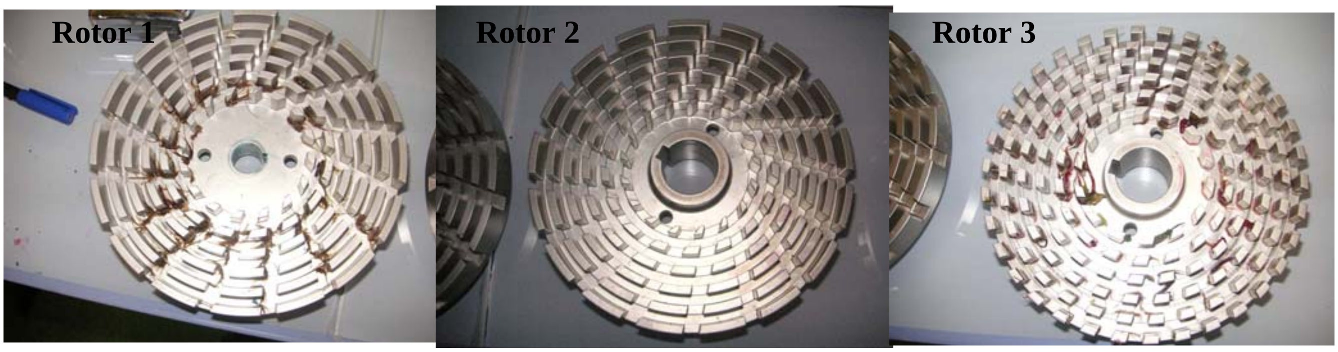


Figure 7. Types of rotor tested in the FRISTAM shear pump.

Figure 8. Stator with drilled teeth tested in the FRISTAM shear pump.

Milling time	0 min	5 min	5 min	5 min	5 min
Stator type		Stator 1	Stator 1	Stator 1	Stator 2
Rotor type		Rotor 1	Rotor 2	Rotor 3	Rotor 2
Average	2,98	1,55	1,63	1,84	1,72
Std dev. (%)	56,96	76,83	66,14	67,42	42,74

Table 4. Particle size (mm) obtained after 5 min of milling with the shear pump with 3 types of rotor and 2 different types of stator (15 particles are measured in each case)

	Fresh	Frozen
Milling time	5 min	5 min
Stator type	Stator 1	Stator 1
Rotor type	Rotor 2	Rotor 2
Average	1,29	1,23
Std dev. (%)	89,71	45,13

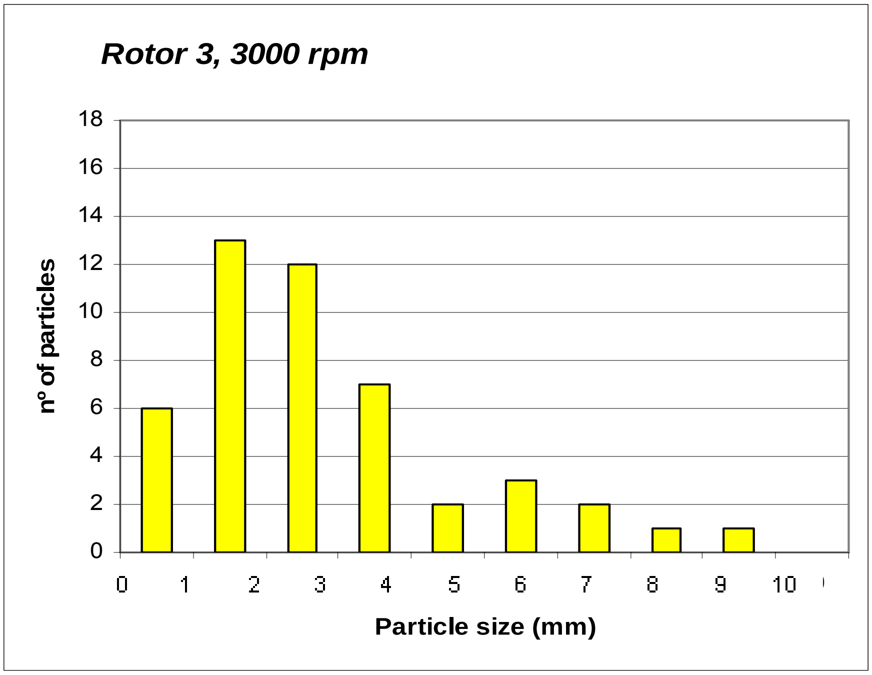
Table 5. Particle size (mm) obtained after 5 min of milling with the shear pump with fresh or frozen pre-grinded vegetables

- The introduction of faeces in the mixture didn´t affect the particle size obtained, and in the case of mixtures including wheat straw, only a higher size and higher dispersion was observed at 5 min of milling

					Mixture including faeces			
	Speed 1		Speed 2		*+ straw*		*+ straw*	
Milling time	5 min	10 min	5 min	10 min	*5 min*	*5min*	*10 min*	*10 min*
Stator type	Stator 1	Stator 1	Stator 1	Stator 1	*Stator 1*	*Stator 1*	*Stator 1*	*Stator 1*
Rotor type	Rotor 3	Rotor 3	Rotor 3	Rotor 3	*Rotor 3*	*Rotor 3*	*Rotor 3*	*Rotor 3*
Average	1,73	2,62	2,22	1,74	2,92	1,79	1,86	1,86
Std dev. (%)	56,06	51,99	39,83	55,51	85,97	39,71	72,94	61,49

Table 6. Particle size (mm) obtained after different time of milling and different speed (Speed 1 = 3000 rpm; Speed 2 = 4000 rpm), with the shear pump in mixtures of beet and lettuce with or without faeces and straw.

As the results obtained with different time of milling and speed were somehow contradictory or at least not significant, and with a high std. deviation as well, additional tests were performed, carrying out a high number of measurements in order to precise better the particle size distribution. The results are shown in Figure 9: a 21% reduction of the average particle size and a change in the size distribution profile was demonstrated when the speed was increased from 3000 to 4000 rpm (average of 50 measurements), and a decrease in the dispersion of the results (from 70 to 51% in std. deviation). However, as the average particle size was still higher than the specified limit of 2 mm, the conclusion was that a more powerful pump providing higher speed (so increasing the shear effect) would be needed for this purpose.



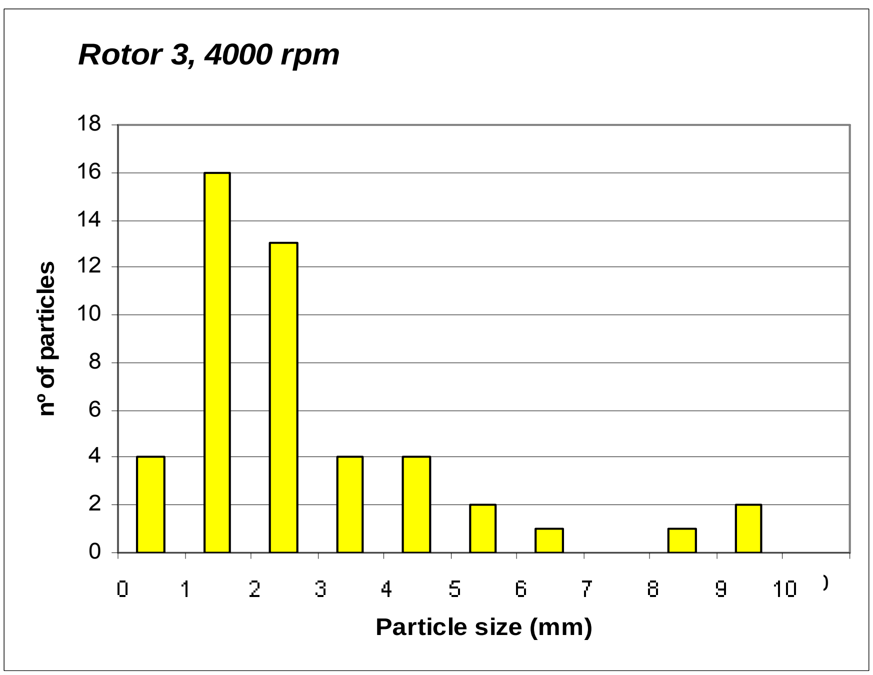


Figure 9. Particle size reduction after 10 min of milling and different speed, with the shear pump in mixtures of lettuce, beet and straw.

4.6. Tests carried out with alternative wet milling systems

Alternative tests were carried out with two additional equipment: a "Cowles" grinding system and an in-line mixer, in both cases performed as well with the vegetable mixture in the concentrations foreseen for the WPU nominal operation conditions.

a) COWLES type grinder (Food Processing Pilot Plant, UAB)

The Cowles system installed in the Food Pilot Plant is an equipment made in-house consisting on an open steel tank with an grinding agitator, by means of a disc impeller equipped with cutting teeth

The existing equipment in the Food Pilot Plant is shown in Figure 10. In such equipment, the big particles of lettuce and beet are reduced in size by means of the impeller. However, many big particles were still remaining after a long time of operation, especially the harder ones (beet), and even more if frozen material was used. This is because the particles are surrounding the impeller but they're not forced enough to be cutted systematically by the impeller. Taking all this into account, the system was discarded as a potential alternative.

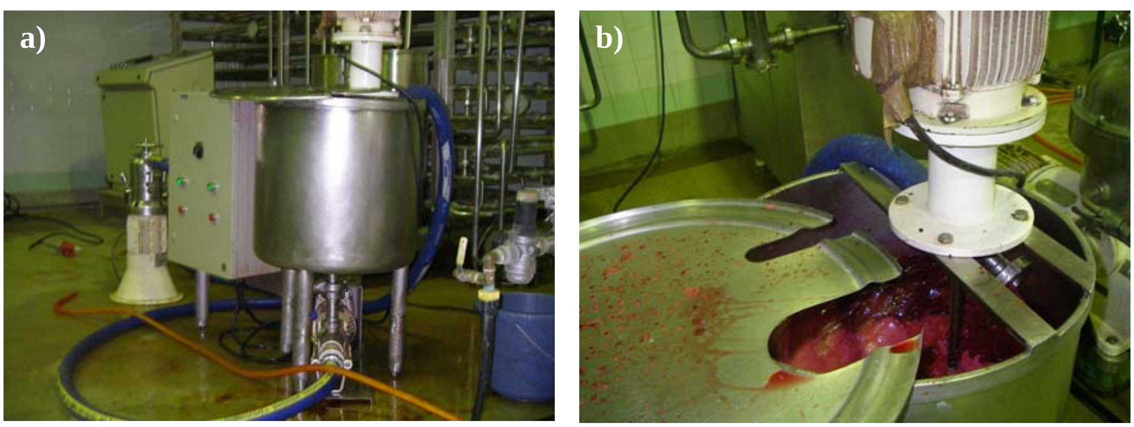


Figure 10. a) In-house made Cowles equipment in the Food Pilot Plant (Veterinary School, UAB) and b) grinding test performed with beet in the same facility.

b) VAK-KIMSA in-line mixer (VAK-KIMSA Pilot Plant, Rubí, Barcelona)

A new test was organized in the VAK-KIMSA Pilot Plant with the in-line mixer VISCOVAK. This equipment is used in a similar design that the FRISTAM shear pump (see Figure 11, a): it is connected in-line with a recirculation pipe and this arrives to a circulation tank (Fig. 11, b). The inlet to the mixer is forced by an additional pump. All the equipment has a sanitary design including clamp connections (Fig. 11, c), and can be CIP and SIP.

Figure 11. a) VISCOVAK in-line mixer and ancillary equipment (VAK KIMSA Pilot Plant, Barcelona); b) fluid recirculation after passing the mixer; c) details of the mixing head; d) detail of non milled particles

The results of this test at 10 min of milling are shown in Figure 12 (average of 50 measurements): the average size of the samples taken from the recycling suspension was lower than 2 mm, and lower as well than the one with the FRISTAM pump. However, the dispersion of the results was higher, and this was due to the fact that some very big particles were not milled, causing the accumulation of the same inside the rotor of the mixing head (Figure 11, c). This dispersion of the measurement was lower at higher times of milling (see Table 7), and a lower average size was obtained. However, the accumulation of long particles within the pump head was still present, making invalid the results. Further engineering would be needed to modify the rotor design in order to cope with this problem. Apart from that, the investment on this equipment is quite high (see Table 3).

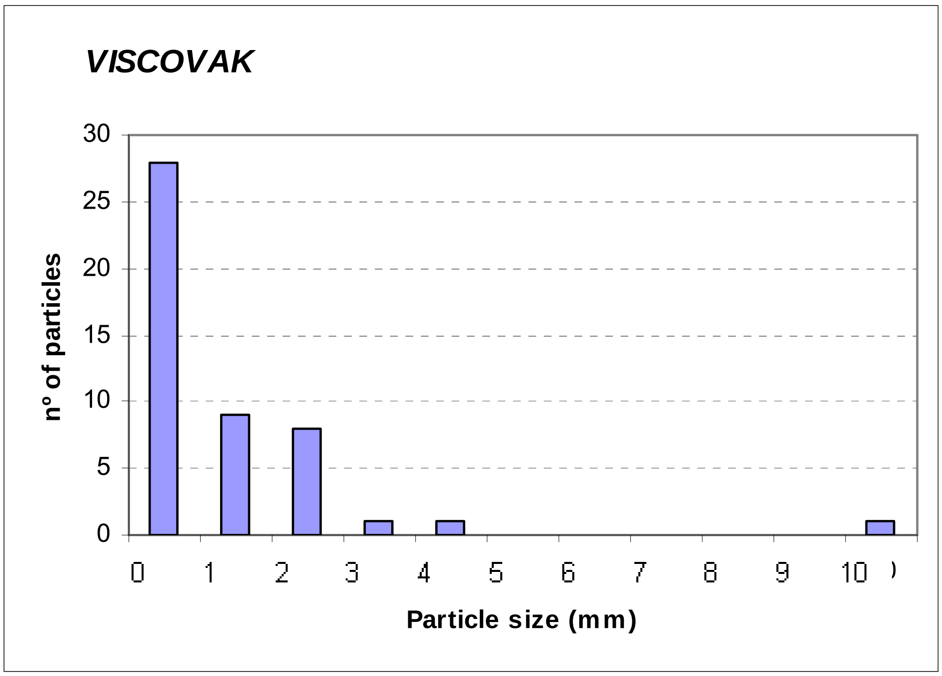


Figure 12. Particle size distribution after 10 min of milling, with the shear pump in a mixture of lettuce, beet and straw

Milling time (min)	6	10	15	20
Average size (mm)	1,56	1,44	1,31	1,31
Std dev. (%)	92,74	117,33	71,60	71,60

Table 7: Particle size and dispersion of the measurements after different times of milling with the VISCOVAK.

4.7. Tests carried out with laboratory mills for straw grinding

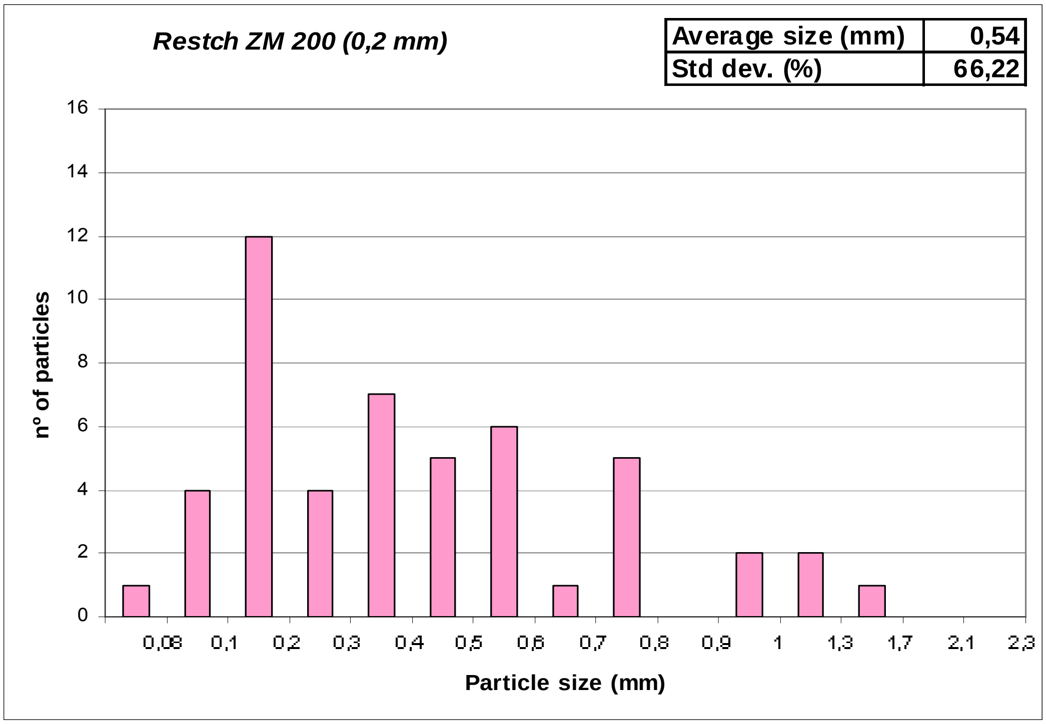
The fist test was performed in RETSCH mobile analytical station with a sample of 50 g of straw. A previous step of grinding using a cutting mill (Model SM 100, Figure 13, a) was performed, in order to obtain previously particles around 3-4 mm in length (4 mm sieve). Then 15g of the product was further grinded by means of the proposed ultra-centrifugal mill (Model ZM-200, Figure 13, b) with a sieve of 0,2 mm hole. A powder 100% passing through a 200 µm sieve was obtained, however, some particles could be still longer than 200 µm, due to the fibrous shape of the straw (see test report in Annex 1).

a)

b)

Figure 13. RETSCH mills: a) Cutting mill SM 100; b) Ultra-centrifugue mill ZM 200

A new test was performed with the cyclone mill Model Cyclotec (0,5 mm screen hole) in the Department of Animal and Food Science (Veterinary Faculty). The sample was previously grinded again by means of a cutting mill SM 100 (available as well in this Department). The results of particle size distribution of the powder obtained, compared with the previous sample milled with the ZM 200, are shown in Figure 14. A similar average size although a higher dispersion of the measurements occurred with the Cyclotec. Nevertheless, it is quite surprising to obtain such a small difference in average between the two equipments considering the big difference in the sieve cut-off (0,5 vs. 0,2). The lack of a precise method for measuring can partially question the significance of these results, and made even more evident the need to find an adequate method for particle size determination.



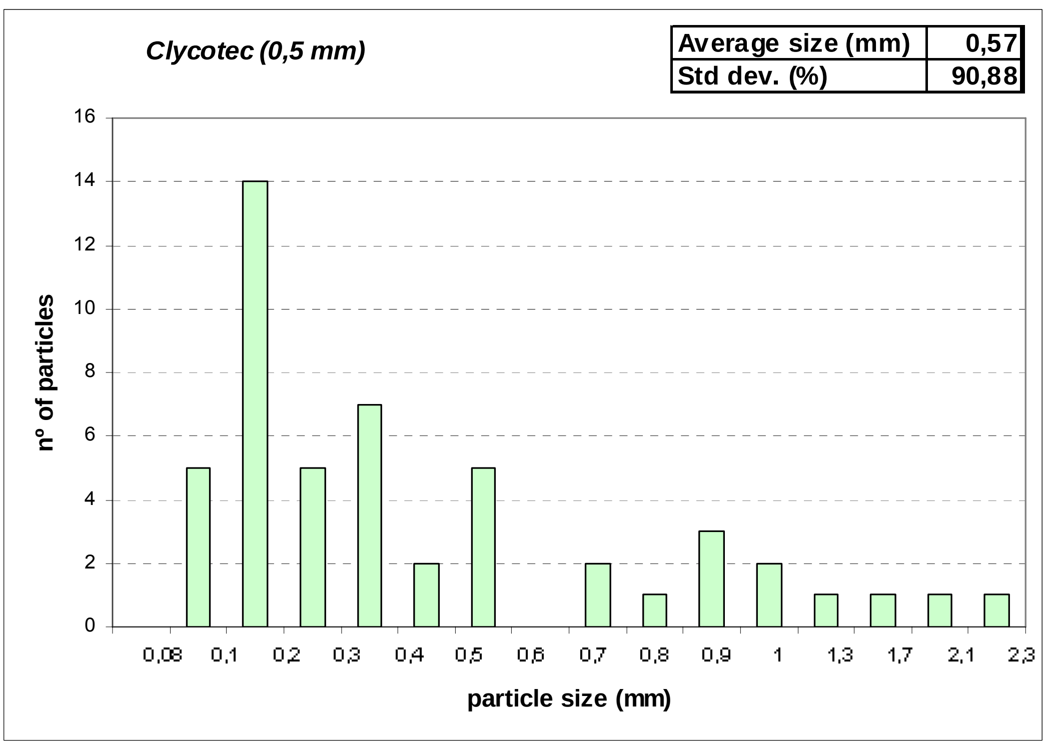


Figure 14. Particle size distribution in straw samples grinded with ultra centrifugal and cyclone mills (microscopic manual measurements)

An additional test was performed with the cutting mill Pulverisette 15 in CREAF (UAB), using a 250 µm mesh. The results are shown in Figure 15. The average size is again quite close to 0,5 mm, but the dispersion of the data is lower in this case.

Taking into account that these results are still far from the objective of 0,2 mm particle size, a new test was performed in RETSCH with the ultra centrifugal mill using a smaller sieve cut-off (0,12 mm). in this case, an alternative test using a Planetary Ball mill was also performed. The results are shown in Figure 16. In this case, a set of sieves was used to evaluate the particle size distribution. As it can be shown, the ultracentrifugal mill was clearly more effective, and 97% of the weight was passing through the 125 µm sieve. However, part of these particles were still longer, even passing through, due to the fibrous shape of the particles, as it has been already discussed.

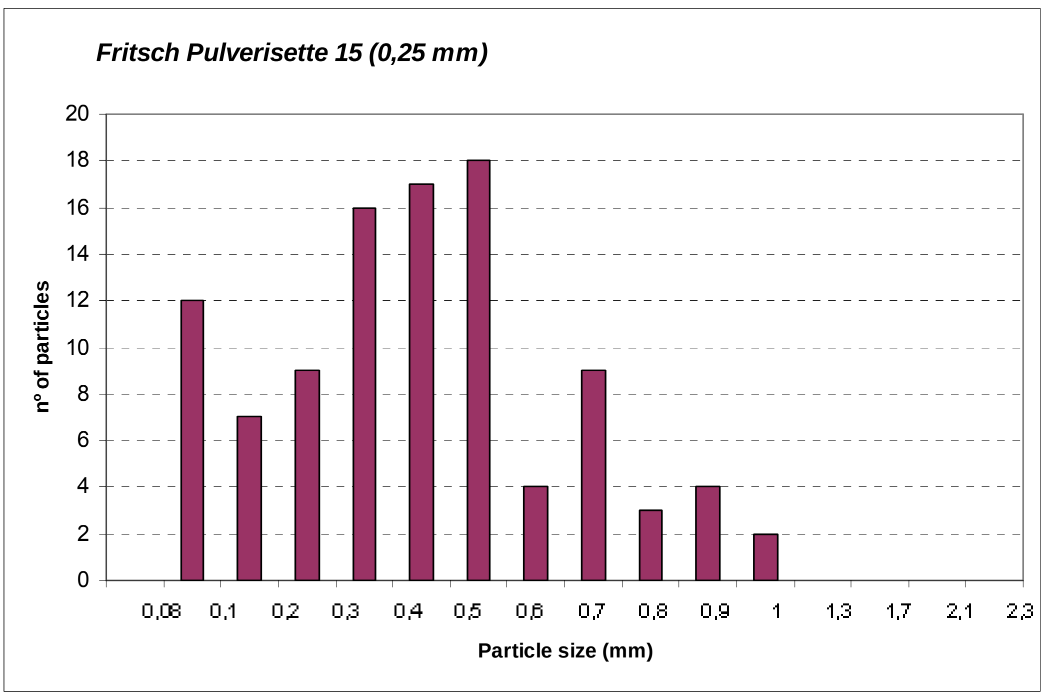
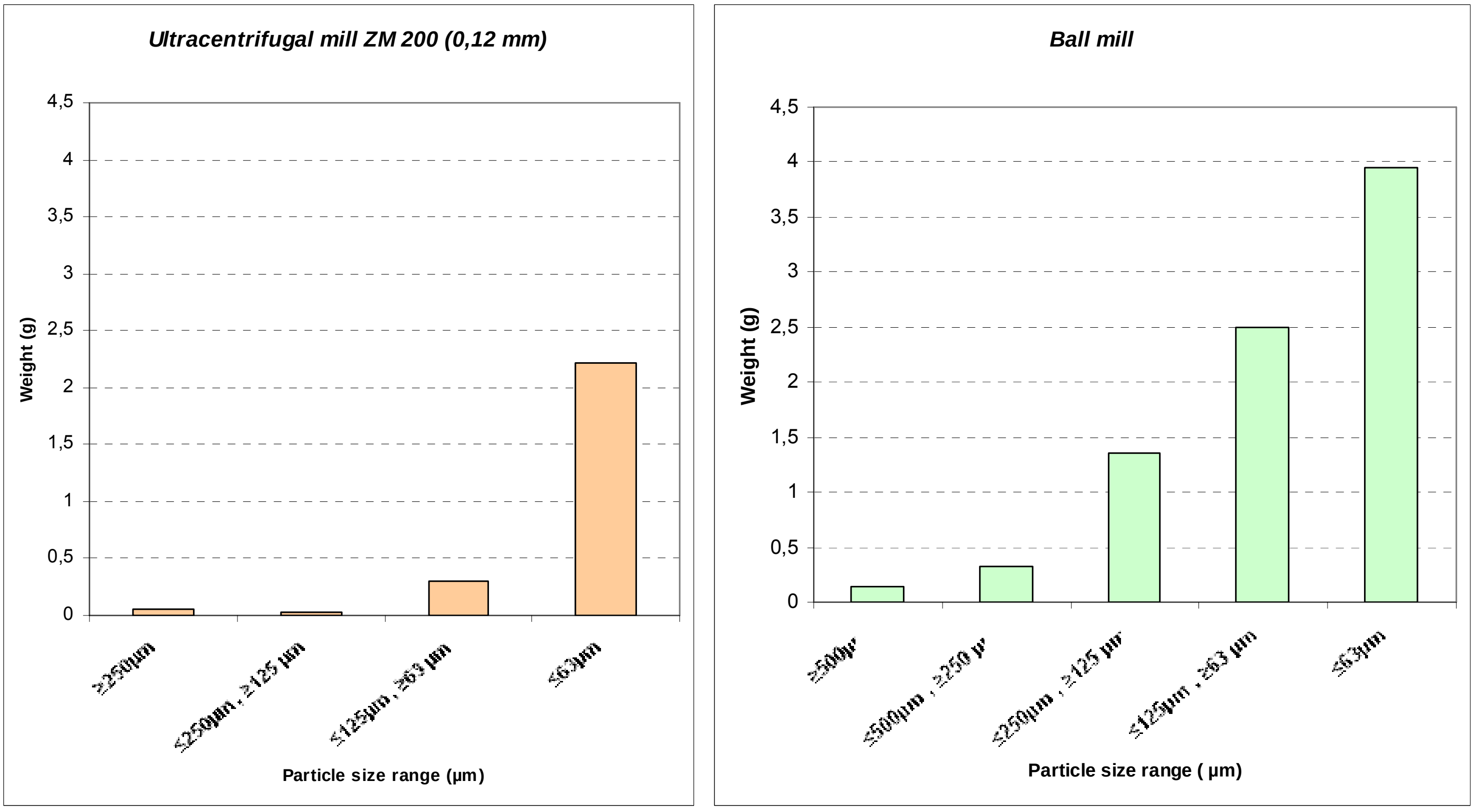


Figure 15. Particle size distribution in straw samples grinded with cutting mill (microscopic manual measurements)



Ultracentrifugal mill		
Particle size range	***Weight (g)***	***% Weight***
≥500µm	0,00	0,00
500-250µm	0,05	1,91
250-125µm	0,02	0,91
125-63µm	0,31	11,78
≤63µm	2,21	85,39

Planetary Ball mill		
Particle size range	***Weight (g)***	***% Weight***
≥500µm	0,14	1,63
500-250µm	0,33	3,94
250-125µm	1,35	16,39
125-63µm	2,49	30,16
≤63µm	3,96	47,87

Figure 16. Particle size distribution in straw samples grinded with ultracentrifugal and ball mills. Total sample weight, 2,59 g and 8,13 g respectively (Measurements by sieving)

4.8. Description of the selected equipment

Taking into account all the results above described both for dry milling (straw) and wet milling of the whole mixture, it was possible to establish the design concept for the WPU in the MELiSSA Pilot Plant (Figure 17).

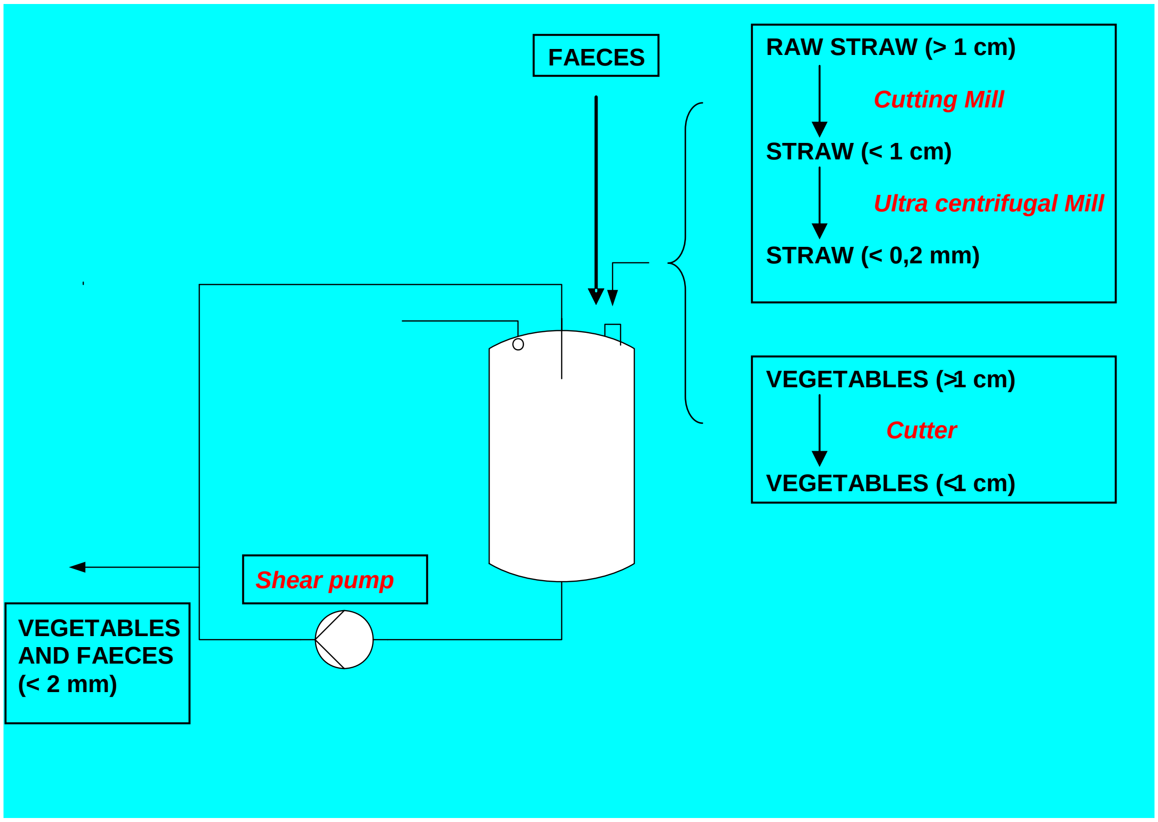


Figure 17: Design concept of the Waste Preparation in the MELiSSA Pilot Plant

The main points on the design are the following:

- Lettuce, beet and toilet paper grinding: two phases are needed, the first one by means of a cutter to obtain particles lower than 1 cm, and a final cutting phase with a shear pump in order to obtain 2mm particles for these vegetables. The selected equipment for the second phase was the FRISTAM shear pump, model of the same foreseen with a higher potency than the one tested, in order to mill at higher speed than 4000 rpm, in order to reduce more the particle size. For the first phase, an industrial food cutter could be used (available in UAB, see section 5).

- Wheat straw grinding: two phases are needed as well, the first one by means of a cutting mill to obtain particles of 3-4 mm size, then a second step of grinding to arrive to 0,2 mm with a ultra centrifugal mill. The selected equipment for the second step was the RETSCH Ultra centrifugal mill ZM-200. For the first phase, there is a Cutting mill available in UAB (see section 5).
- Faeces grinding and overall mixing: this operation coincides with the second phase of the vegetables grinding. The faeces and the milled straw are mixed with the pre-cutted vegetables and paper in the recirculation tank, and the whole mixture is passed through the shear pump, in order to provide 2 mm particle size as the minimum, compatible with 0,2 in the case of the straw (previously obtained).

Further tests including precise particle size measurements were needed in order to take final decisions on the investments on the shear pump and the ultra centrifugal mill. In the case of the shear pump, a step-wise approach involving renting was decided, in order to test the whole preparation unit, where the pump acts both as a grinding equipment but as well as the mixing element. In the case of the ultra centrifugal mill, the performance of more precise measurements was considered mandatory before taking a final decision.

5. DEFINITION OF THE WASTE PRETREATMENT PHASE

The available equipment existing in UAB was taken into consideration, both to reduce the investment needed and as well the manpower costs. Agreements with two departments were achieved: the Centre Especial de Recerca Planta de Tecnologia dels Aliments (Food Processing Technology Plant) and the Departament de Ciència Animal i dels Aliments (Animal and Food Science), both in UAB Veterinary Faculty.

Food Technology Plant: it provides the following services:

a) Lettuce and beet reception in the Food Technology Plant facility from the freezing warehouse, and straw and toilet paper from the MPP.
b) Refrigerated storage of raw materials up to their preparation for milling
c) Raw materials weighing and mixture preparation
d) Cutting in an industrial kitchen cutter (TEC MAQ Mod. CUT-20, INTERFI, S.A., 25 Kg capacity)
e) Delivery of the grinded mixture to the MPP.

Animal and Food Science Department: it provides the following services:
a) Straw reception from the MPP.
b) Room temperature storage of straw up to the preparation of the same for milling
c) Straw weighing

d) Straw milling with a cutting mill (Mod. SM 100, RETSCH)
e) Delivery of the grinded straw to the MPP.

6. PARTICLE SIZE STUDIES

6.1. Selection of an off-line particle size determination method

Several Departments in UAB and companies involved in particle size measurement were contacted, in order to implement an adequate method for particle size measurements both for the vegetables and straw, with the aim of validate the WPU selected design.

The equipment that was selected for the measuring tests was the Mastersizer S (MALVERN INSTRUMENTS Ltd., England), being able of measure particle size ranges up to 3 mm (see commercial info in Annex 2 –the equipment there described ranges only up to 2 mm-). This equipment is installed in the Centro de Ciencias Medioambientales (CSIC, Madrid).

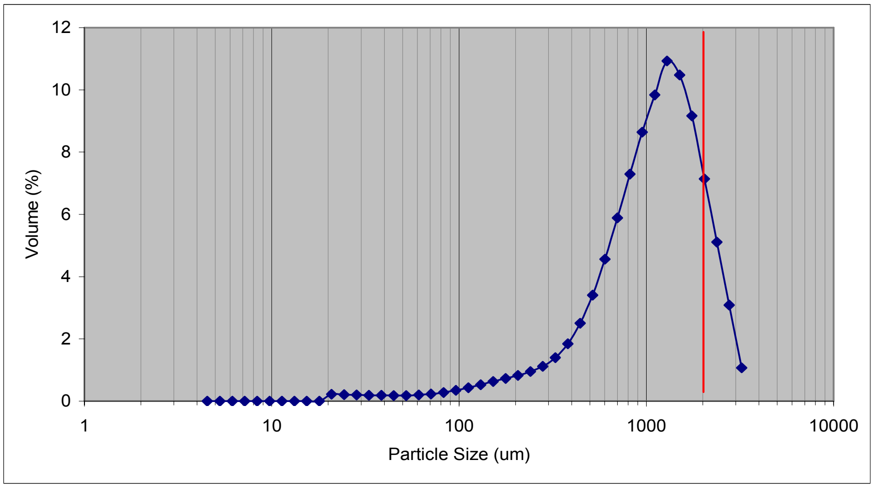
6.2. Analysis of real samples from WPU output

In a first approach, three samples were sent to be measured: Pre-milled Feed in an industrial cutter, Wheat straw after milling with the Cyclotec (0,5 mm mesh) and Wheat straw after milling with the Pulverisette 15 (0,25 mm mesh). There wasn't any sample left obtained with the Restch ultracentrifugal mill to be analysed at that time.

The samples were measured with a Mastersizer S, red laser, wavelength 633 nm, by Fraunhofer and Mie method in wet, lens 1000 mm (range 4 µm – 3500 µm) in water plus a dispersant (Nonidet P40). In the Feed sample, 3 measurements of 10.000 readings were performed in 20 seconds, and in Straw samples, 6 measurements (in twice) of 10.000 readings were performed in 20 seconds. The average of those readings was calculated and is shown in the graphics.

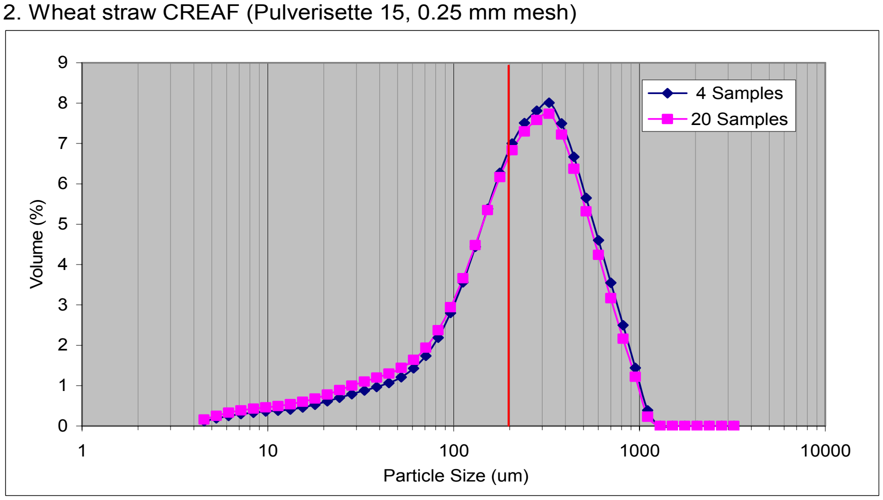
The results are summarized in Figure 18 and all the methodology details are described in Annex 3. In the case of the feed sample, it can be deduced that already 91% of the volume of particles are compliant with the objective of 2 mm particle size. This is relevant also because only a small percentage is supposed not to have been measured within the measuring range of the equipment (<3,5 mm), only the right end of the curve is missing.

1. FEED lot number: 13 (04.04.09)



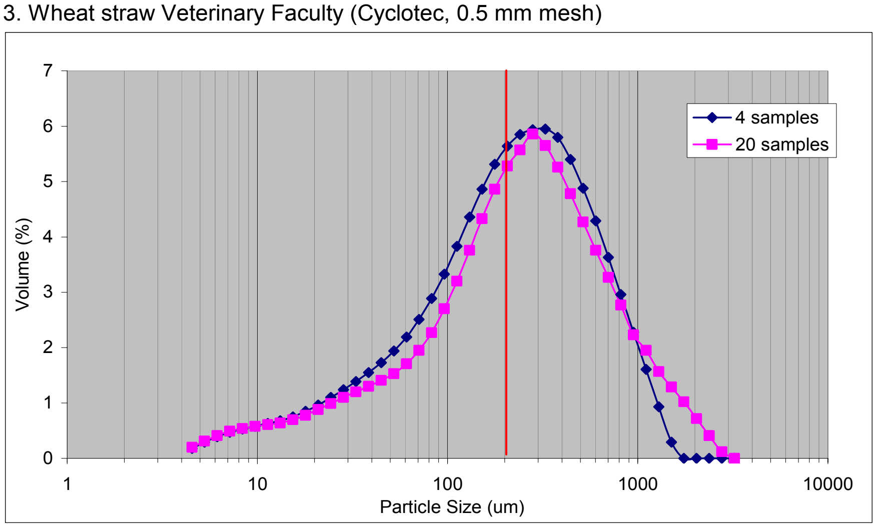
Target size	2000	um
Volume of particles ≤ the target size	**91,0**	%

2. Wheat straw CREAF (Pulverisette 15, 0.25 mm mesh)



Target size	200	um
Volume of particles ≤ the target size	**44,1**	%

3. Wheat straw Veterinary Faculty (Cyclotec, 0.5 mm mesh)



Target size	200	um
Volume of particles ≤ the target size	**49,9**	%

Figure 18: Particle size distribution of Feed and Straw samples measured with MASTERSIZER S

However, the values obtained for the milled straw are far from the objective of 0,2 mm particle size: in the case of the Cyclotec, approx. 50% of the volume of particles were compliant with the specification (<0,2 mm), and for the Pulverisette this value was only 44%, in spite of a much smaller mesh size. This may indicate a better performance of the ultracentrifugal mill respect to the cutting mill. The results as well show that the estimation of measurements made manually were not so far from the real situation.

Once the WPU was installed in the MPP including the FRISTAM pump (Mod. FSPE 3522, 11 Kw), a new test was performed in order to take several samples of CI Feed mixture along the milling time with the shear pump. The samples were again sent to the Centro de Ciencias Medioambientales (CSIC). The results are shown in Annex 4. The samples were again measured with the Mastersizer S with the method already described. In all samples, 4 measurements of 1.000 readings were performed in 10 seconds, the average of those readings being calculated and shown in the graphics. In Table 8 the % volume compliant with the specification of 2 mm is shown for each sample along the milling time (always considering discarded any volume of particles higher than 3,5 mm, not measured by this instrument) :

Milling time (min)	1	3	5	10	15	20
% Vol. <2 mm	86,1	87,8	89,0	89,9	91,2	92,3

Table 8. %Vol. < 2mm in the Feed samples during the milling operation with the FRISTAM pump.

As it can be deduced from these data and from the graphs, the main effect obtained by the shear pump was within the first minutes of milling, being the rest up to 20 min only slightly effective.

Further tests should be performed in order to assess more precisely the influence of changes in the speed of milling, recirculation flows or other factors in the operation of the shear pump in order to comply with the size specification.

Regarding the straw milling, the results show the need of improving the milling/sieving technique in order to comply with the more stringent specification.

7. CONCLUSIONS

In the frame of this technical note, the design conpcept of the WPU within the MPP has been described, and the trade-off among different equipments available in the market has been carried out; the equipment used in EPAS has been as well evaluated, and the potentiality of using external services in UAB in order to reduce the scope of investment has been addressed. As a result, a combined system involving a pre-treatment phase and a final milling phase has been defined, and a separated way of milling (dry basis for straw; wet basis for vegetables and total mix) has been selected.

The work performed involves as well the selection of the particle size measurement technique involving microscopic measurements, sieving and laser diffraction (light scattering).

The results obtained show the difficulty to obtain 100% vegetable particle sizes lower than 2 mm in the feeding mix of CI, and the effectivity is still much lower regarding the straw, where only about 50% of the particles in the analysed conditions are compliant with the 0,2 mm objective.

Several proposals for the completion of the current trade-off tasks should be here considered:

- To perform new tests on the WPU during its validation, in order to do particle size measurements (light scattering) of the wet milling vs. changes in the speed of milling, recirculation flows or other factors in the operation of the shear pump in order to comply with the size specification.
- To perform new tests with the Ultracentrifugal mill using 0,12 and 0,2 mm ring sieves, and take samples for particle size determination with the same equipment above mentioned.
- To evaluate the obtained measurements vs. the specifications in both wet and dry milling conditions, the existing specifications needing to be revisited taking into account not only the specified threshold sizes for straw and vegetables (0,2 and 2 mm respectively) but as well potential acceptance criteria based on particle size distribution or % of compliant volume of particles, coherent with the needs of CI compartment.

Annex 1

Test Report.

Retsch®

Report No.: **11717**
Date: 21.5.2007
Contact: ATH

Task:

Application field:	Agriculture
Material:	**Wheat straw**
Feed size:	**length: 100 mm (before pre-grinding),** **30 mm (before fine grinding)**
Feed quantity:	50 g (pre-grinding), 15 g (fine grinding)
Material specification(s):	fibrous
Customer requirement(s):	100 % < 200 µm (SM 100 available, fine grinding in ZM 200 with cyclone)
Subsequent analysis:	Digestion in pilot plant

Solution:

Selected instrument(s):	**Cutting Mill SM 100** **Ultra Centrifugal Mill ZM 200**
Configuration(s):	SM 100: Bottom sieve square holes 4 mm, stainless steel; ZM 200: Push-fit rotor, 12 teeth, stainless steel; Ring sieve conidur holes 0.2 mm, stainless steel
Parameter(s):	SM 100: approx. 1400 rpm ZM 200: 18000 rpm
Time:	30 s (pre-grinding), 10 s (fine grinding)
Achieved result(s):	100 % < 200 µm (the length of some particles can be > 200 µm)
Remark(s):	For grinding of larger quantities a cyclone can be used. (not tested, due to the small sample quantity)
Recommendation:	For pre-grinding the Cutting Mill SM 100 and for fine grinding the Ultra Centrifugal Mill ZM 200 are suitable under the above mentioned conditions.

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page 1/2

Test Report.

Retsch®

Report No.: **11717**
Date: 21.5.2007
Contact: ATH

Pictures of the sample

Fig. 1: after pre-grinding in SM 100 with 4 mm bottom sieve

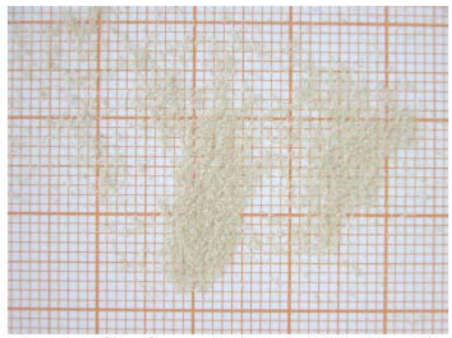


Fig. 2: after fine grinding in ZM 200 with 0.2 mm ring sieve

Annex 2

Mastersizer 2000

"Routine use of Standard Operating Procedures and minimal maintenance requirements associated with the Mastersizer 2000 are essential in plant laboratories. At the same time, however, the flexibility of the Mastersizer 2000 software and its wide measurement range are important in R&D." (OMYA AG)

The Mastersizer 2000 particle size analyzer has been developed to meet industry's growing need for global comparability of results, traceability, regulatory compliance, and efficiency in the laboratory. In introducing the Mastersizer 2000 particle size analyzer, Malvern has advanced particle size analysis to the point where it is now a simple, straightforward and routine task.

- **Accuracy and Reproducibility**

Accuracy: ± 1% on the Dv50 using the Malvern Quality Audit Standard. Instrument-to-instrument reproducibility: Better than 1% RSD on the Dv50 using the Malvern Quality Audit Standard.

- **Assured reproducibility**

Software-driven SOPs eliminate user variability and enable global method transfer. All measurement parameters are automatically embedded in the result files and can be critically reviewed by e-mail recipients. Measurements can be replicated by following the same SOP.

- **Broad measuring range**

Measures materials from 0.02µm to 2000µm.

- **Wide range of sample types**

For the measurement of emulsions, suspensions and dry powders.

- **Ease of use**

Fully automated for ease of use. Designed to eliminate user-to-user variability. Reduces training requirements for new users and maximizes deployment of skilled personnel.

- **Flexibility**

Wide range of sample dispersion units. Rapid changeover between sample dispersion units with automatic configuration. "Plug and Play" cassette system allows simultaneous connection of two sample dispersion units.

- **Regulatory Compliance**

Full QSpec validation documentation available including compliance with 21 CFR Part 11.

- **User-friendly Software**

Software-driven Standard Operating Procedures (SOPs) eliminate user variability.
An SOP-creation wizard helps users to create good measurement methods.
On-line help and advice on every aspect of performing a measurement available at the click of a button
On screen instructions lead user through the measurement process.
SOPs supplied for routine sample analyzes.
Custom Report Designer allows screen layout and printouts to be configured to suit each user.
Custom calculator allows the parameters important to your application to be defined.

Mastersizer 2000 technical specifications

Optical Unit	Specification	
Size range	Materials in the range 0.02µm to 2000µm	
Measurement principle	Mie scattering	
Detection systems	Red light: forward scattering, side scattering, back scattering Blue light: wide angle forward and back scattering	
Light sources	Red light: helium-neon laser Blue light: solid-state light source	
Optical alignment system	Automatic rapid align system with dark field optical reticle	
Sample dispersion unit interchange	Sample dispersion units automaticaly recognized, configured and enabled on insertion of measurement cell cassettes into sizer	
Laser system	Mastersizer 2000: Class 1 laser product Autosampler 2000: Class 2 laser product LASER RADIATION DO NOT STARE INTO BEAM CLASS 2 LASER PRODUCT	
Software and data processing		
Minimum Computer Specification	IBM compatible PC Pentium 166MHz, 32MByte RAM (64MByte recommended) and CD-ROM. SVGA screen with 800 x 600 resolution, 256 colour. At least 100MByte of free hard disk space is required to operate the software. This specification does not take into account the operating system requirements. Please note:The MS2000 Autosampler requires 128MByte of free hard disk space and a 1024 x 768 screen resolution.	
Operating Systems	Windows NT v 4.0 (Service Pack 6A or Higher), Windows 2000 Professional (Service Pack 2 or Higher) or Windows XP Professional. Windows 2000 Professional is the recommended operating system.	
Database utility	Searching, sorting and filtering by search criteria of data records on all parameters of interest.	
Custom report facility	Custom report designer using drag-and-drop selection, positioning and sizing of key report elements.	
Creation of SOPs and automation	Set up by means of SOP Wizard with extensive advice at all stages of SOP creation. A library of SOPs for common materials is built into the software as standard.	
Operating modes	Automated using SOPs created in the software. Manual, using on-screen controls and hot keys.	
Weights and dimensions		
Model	**Unpacked weight (kg)**	**Dimensions (length; depth; height in mm)**
Mastersizer 2000 optical bench	31.0	1293 x 255 x 375
Hydro 2000G	13.7	344 x 352 x 330
Hydro 2000S	11.0	352 x 355 x 332
Hydro 2000MU	15.4	320 x 375 x 335/490
Hydro 2000 Micro Precision	12.2	287 x 253 x 338
Scirocco 2000	11.7	352 x 355 x 332
Autosampler 2000	32.0	550 x 365 x 560

11 Mastersizer 2000 www.malvern.co.uk

Annex 3

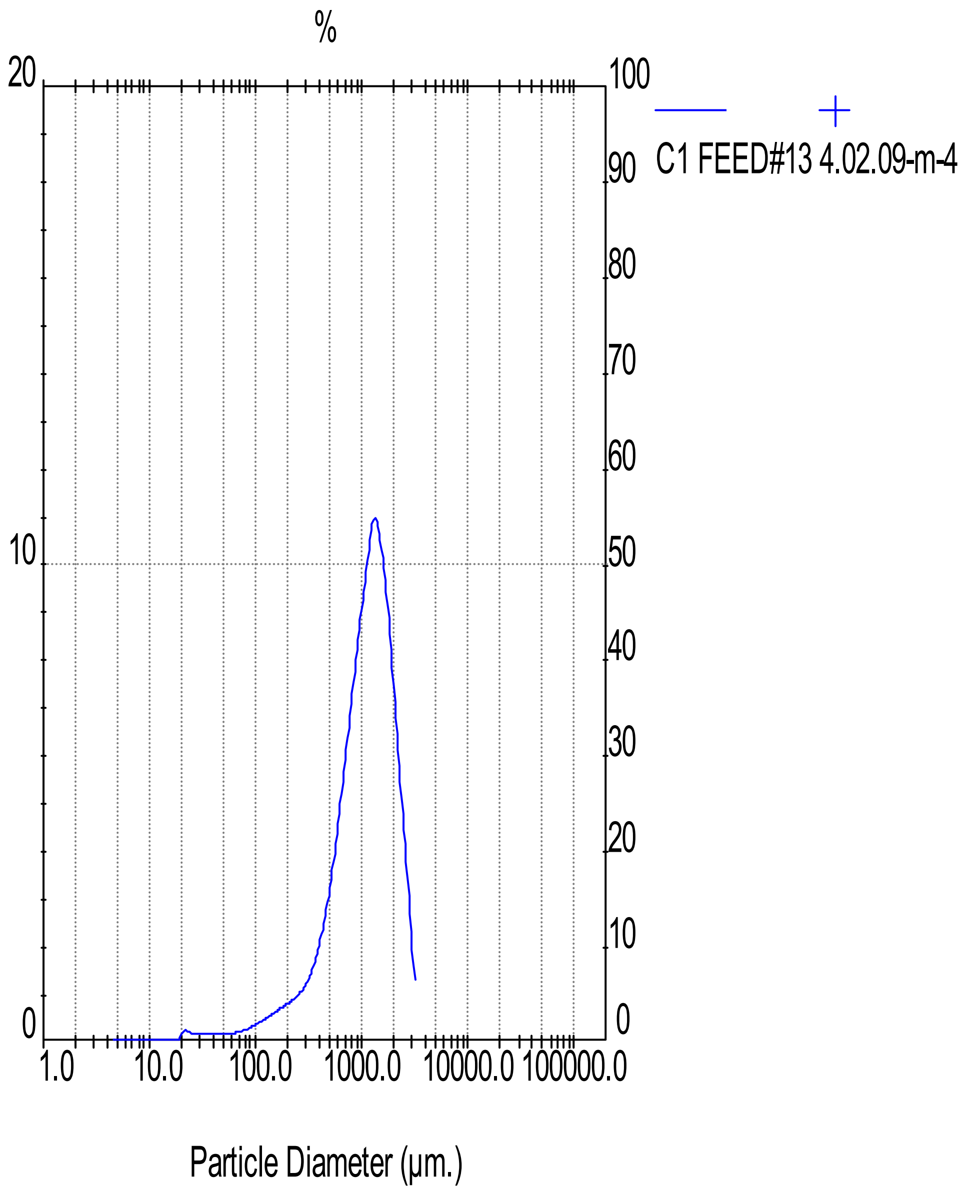
Result: Analysis Table

ID: C1 FEED#13 4.02.09-m	Run No: 4	Measured:
File: PEIRO	Rec. No: 12	Analysed: 6/3/2009 16:24
Path: K:\		Source: Averaged

Range: 1000 mm	Beam: 2.40 mm	Sampler:	Obs': 12.5 %
Presentation: 3OHD		Analysis: Polydisperse	Residual: 4.213 %
Modifications: None			

Conc. = 1.1257 %Vol	Density = 1.000 g/cm^3	S.S.A.= 0.0107 m^2/g
Distribution: Volume	D[4, 3] = 1214.52 um	D[3, 2] = 559.75 um
D(v, 0.1) = 382.51 um	D(v, 0.5) = 1140.22 um	D(v, 0.9) = 2157.95 um
Span = 1.557E+00	Uniformity = 4.757E-01	

Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %
4.19	0.00	22.49	0.21	120.67	0.53	647.41	5.89
4.88	0.00	26.20	0.20	140.58	0.63	754.23	7.29
5.69	0.00	30.53	0.19	163.77	0.73	878.67	8.64
6.63	0.00	35.56	0.19	190.80	0.83	1023.66	9.84
7.72	0.00	41.43	0.18	222.28	0.95	1192.56	10.93
9.00	0.00	48.27	0.18	258.95	1.12	1389.33	10.48
10.48	0.00	56.23	0.20	301.68	1.40	1618.57	9.16
12.21	0.00	65.51	0.23	351.46	1.84	1885.64	7.14
14.22	0.00	76.32	0.28	409.45	2.50	2196.77	5.11
16.57	0.00	88.91	0.35	477.01	3.41	2559.23	3.09
19.31	0.22	103.58	0.43	555.71	4.56	2981.51	1.07
22.49		120.67		647.41		3473.45	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Particle Diameter (µm.)
C1 FEED#13 4.02.09-m-4

Result: Analysis Table

ID: Paja Creaf-m	Run No: 4	Measured:
File: PEIRO	Rec. No: 4	Analysed: 6/3/2009 15:58
Path: K:\		Source: Averaged

Range: 1000 mm	Beam: 2.40 mm	Sampler:	Obs': 16.7 %
Presentation: 3OHD		Analysis: Polydisperse	Residual: 0.754 %
Modifications: None			

Conc. = 0.2581 %Vol	Density = 1.000 g/cm^3	S.S.A.= 0.0573 m^2/g
Distribution: Volume	D[4, 3] = 296.36 um	D[3, 2] = 104.78 um
D(v, 0.1) = 59.06 um	D(v, 0.5) = 249.43 um	D(v, 0.9) = 600.80 um
Span = 2.172E+00	Uniformity = 6.589E-01	

Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %
4.19	0.12	22.49	0.70	120.67	4.44	647.41	3.55
4.88	0.19	26.20	0.79	140.58	5.38	754.23	2.50
5.69	0.25	30.53	0.88	163.77	6.27	878.67	1.44
6.63	0.30	35.56	0.97	190.80	7.00	1023.66	0.39
7.72	0.33	41.43	1.07	222.28	7.51	1192.56	0.00
9.00	0.36	48.27	1.21	258.95	7.81	1389.33	0.00
10.48	0.38	56.23	1.43	301.68	8.01	1618.57	0.00
12.21	0.41	65.51	1.74	351.46	7.50	1885.64	0.00
14.22	0.46	76.32	2.19	409.45	6.67	2196.77	0.00
16.57	0.53	88.91	2.80	477.01	5.65	2559.23	0.00
19.31	0.61	103.58	3.56	555.71	4.60	2981.51	0.00
22.49		120.67		647.41		3473.45	

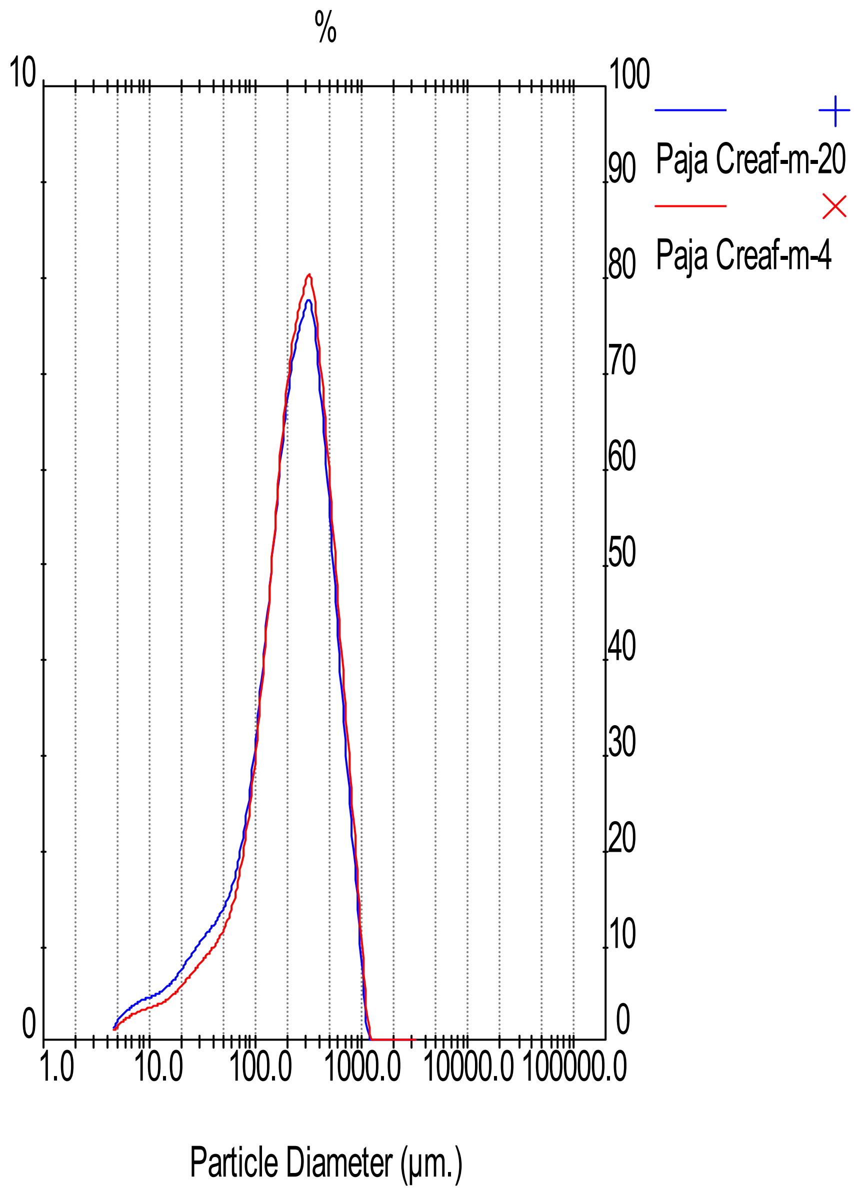
Result: Analysis Table

ID: Paja Creaf-m	Run No: 20	Measured:
File: PEIRO	Rec. No: 15	Analysed: 16/3/2009 11:09
Path: K:\		Source: Averaged

Range: 1000 mm	Beam: 2.40 mm	Sampler:	Obs': 17.4 %
Presentation: 3OHD		Analysis: Polydisperse	Residual: 0.902 %
Modifications: None			

Conc. = 0.2329 %Vol	Density = 1.000 g/cm^3	S.S.A.= 0.0665 m^2/g
Distribution: Volume	D[4, 3] = 279.58 um	D[3, 2] = 90.17 um
D(v, 0.1) = 44.93 um	D(v, 0.5) = 234.67 um	D(v, 0.9) = 574.64 um
Span = 2.257E+00	Uniformity = 6.846E-01	

Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %
4.19	0.16	22.49	0.89	120.67	4.48	647.41	3.17
4.88	0.25	26.20	1.00	140.58	5.35	754.23	2.16
5.69	0.33	30.53	1.10	163.77	6.17	878.67	1.22
6.63	0.39	35.56	1.20	190.80	6.83	1023.66	0.23
7.72	0.43	41.43	1.30	222.28	7.30	1192.56	0.00
9.00	0.46	48.27	1.44	258.95	7.58	1389.33	0.00
10.48	0.49	56.23	1.64	301.68	7.74	1618.57	0.00
12.21	0.54	65.51	1.94	351.46	7.22	1885.64	0.00
14.22	0.60	76.32	2.37	409.45	6.37	2196.77	0.00
16.57	0.68	88.91	2.94	477.01	5.32	2559.23	0.00
19.31	0.78	103.58	3.66	555.71	4.24	2981.51	0.00
22.49		120.67		647.41		3473.45	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Particle Diameter (µm.)
Paja Creaf-m-20
Paja Creaf-m-4

Result: Analysis Table

ID: 018/09 3/3Paja05/0-m   Run No: 4   Measured:
File: PEIRO   Rec. No: 8   Analysed: 6/3/2009 16:13
Path: K:\   Source: Averaged

Range: 1000 mm   Beam: 2.40 mm   Sampler: Obs': 23.3 %
Presentation: 3OHD   Analysis: Polydisperse   Residual: 0.656 %
Modifications: None

Conc. = 0.2756 %Vol   Density = 1.000 g/cm^3   S.S.A.= 0.0759 m^2/g
Distribution: Volume   D[4, 3] = 303.34 um   D[3, 2] = 79.02 um
D(v, 0.1) = 35.42 um   D(v, 0.5) = 221.26 um   D(v, 0.9) = 693.40 um
Span = 2.974E+00   Uniformity = 9.109E-01

Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %
4.19	0.18	22.49	1.10	120.67	4.36	647.41	3.63
4.88	0.29	26.20	1.24	140.58	4.86	754.23	2.96
5.69	0.39	30.53	1.39	163.77	5.31	878.67	2.28
6.63	0.47	35.56	1.55	190.80	5.64	1023.66	1.61
7.72	0.53	41.43	1.73	222.28	5.85	1192.56	0.93
9.00	0.58	48.27	1.94	258.95	5.94	1389.33	0.29
10.48	0.63	56.23	2.19	301.68	5.95	1618.57	0.00
12.21	0.68	65.51	2.51	351.46	5.80	1885.64	0.00
14.22	0.75	76.32	2.89	409.45	5.40	2196.77	0.00
16.57	0.85	88.91	3.33	477.01	4.88	2559.23	0.00
19.31	0.96	103.58	3.83	555.71	4.29	2981.51	0.00
22.49		120.67		647.41		3473.45	

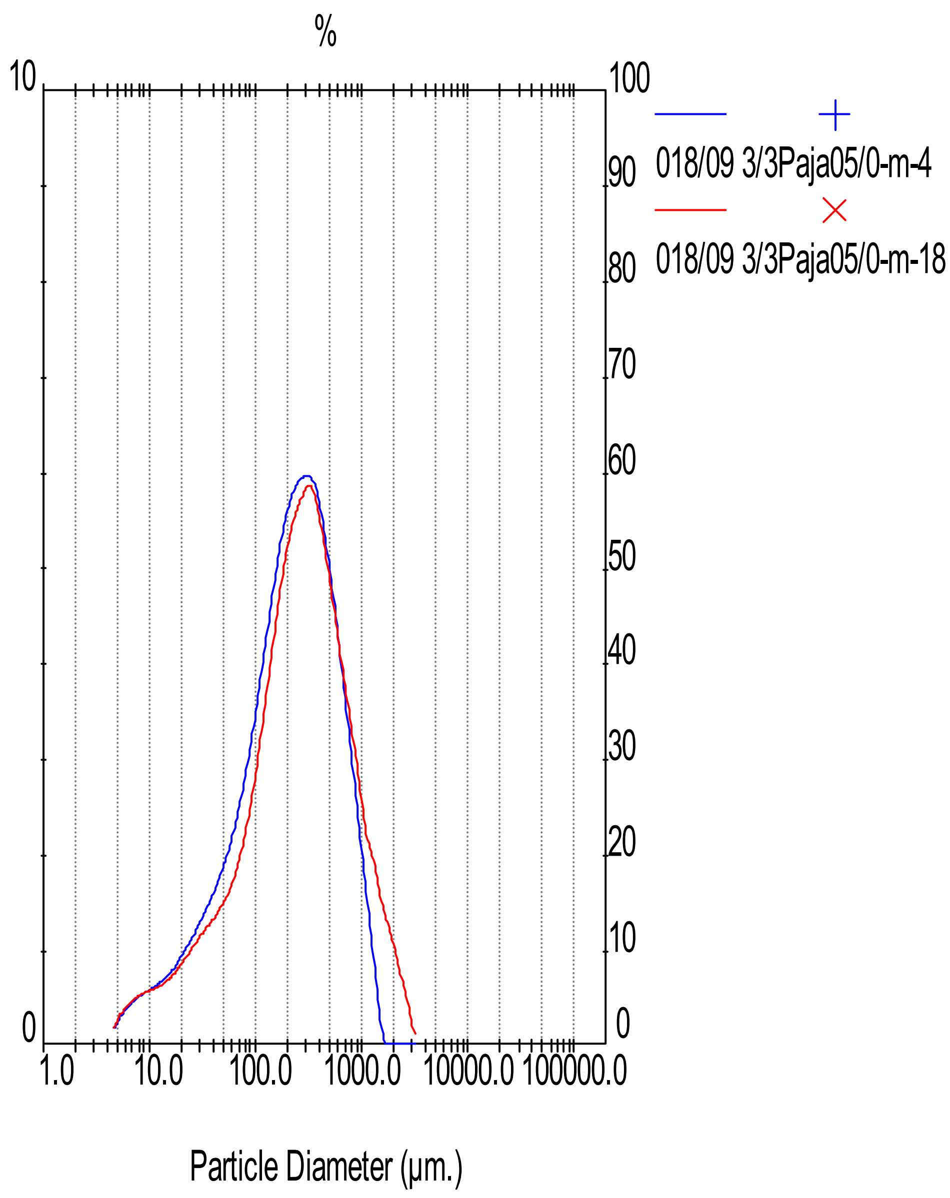
Result: Analysis Table

ID: 018/09 3/3Paja05/0-m   Run No: 18   Measured:
File: PEIRO   Rec. No: 23   Analysed: 16/3/2009 14:24
Path: K:\   Source: Averaged

Range: 1000 mm   Beam: 2.40 mm   Sampler: Obs': 17.3 %
Presentation: 3OHD   Analysis: Polydisperse   Residual: 0.727 %
Modifications: None

Conc. = 0.1659 %Vol   Density = 1.000 g/cm^3   S.S.A.= 0.0710 m^2/g
Distribution: Volume   D[4, 3] = 416.30 um   D[3, 2] = 84.54 um
D(v, 0.1) = 37.99 um   D(v, 0.5) = 263.71 um   D(v, 0.9) = 982.10 um
Span = 3.580E+00   Uniformity = 1.122E+00

Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %
4.19	0.20	22.49	0.99	120.67	3.76	647.41	3.76
4.88	0.31	26.20	1.10	140.58	4.33	754.23	3.27
5.69	0.41	30.53	1.20	163.77	4.86	878.67	2.77
6.63	0.49	35.56	1.30	190.80	5.28	1023.66	2.23
7.72	0.54	41.43	1.41	222.28	5.57	1192.56	1.95
9.00	0.58	48.27	1.53	258.95	5.75	1389.33	1.57
10.48	0.61	56.23	1.71	301.68	5.86	1618.57	1.29
12.21	0.64	65.51	1.95	351.46	5.65	1885.64	1.02
14.22	0.70	76.32	2.27	409.45	5.26	2196.77	0.72
16.57	0.78	88.91	2.70	477.01	4.78	2559.23	0.41
19.31	0.88	103.58	3.20	555.71	4.27	2981.51	0.12
22.49		120.67		647.41		3473.45	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Particle Diameter (µm.)
018/09 3/3Paja05/0-m-4
018/09 3/3Paja05/0-m-18

Annex 4

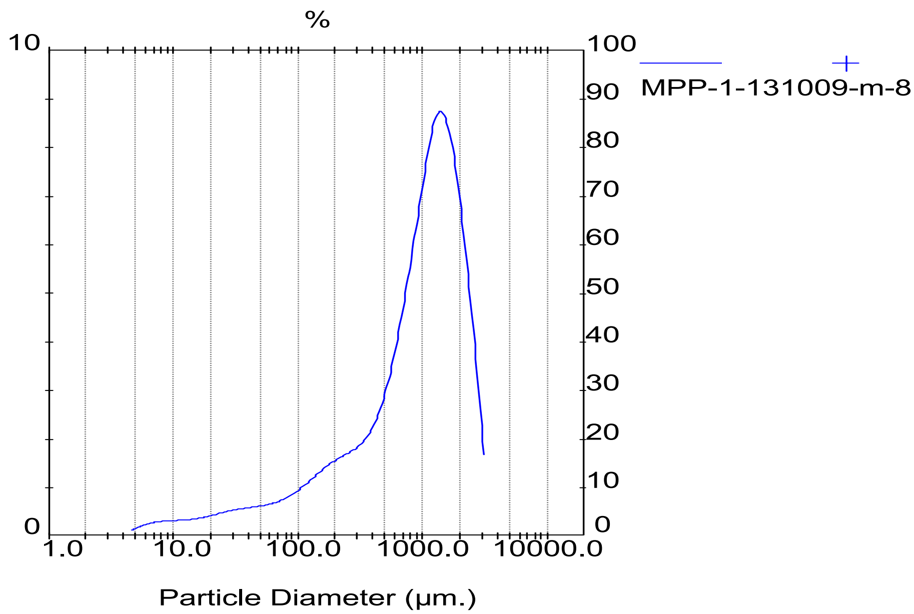
Result: Histogram Table

ID: MPP-1-131009-m	Run No: 8	Measured:
File: 2PEIRO~1	Rec. No: 36	Analysed: 14/10/2009 12:00
Path: K:\		Source: Averaged

Range: 1000 mm	Beam: 2.40 mm	Sampler:	Obs': 13.1 %
Presentation: 3OHD		Analysis: Polydisperse	Residual: 3.116 %
Modifications: None			

Conc. = 0.2878 %Vol	Density = 1.000 g/cm^3	S.S.A.= 0.0366 m^2/g
Distribution: Volume	D[4, 3] = 1093.09 um	D[3, 2] = 163.84 um
D(v, 0.1) = 106.30 um	D(v, 0.5) = 1016.48 um	D(v, 0.9) = 2190.45 um
Span = 2.050E+00	Uniformity = 6.290E-01	

Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %
4.19	0.05	22.49	0.21	120.7	0.50	647.4	1.94
4.48	0.05	24.05	0.21	129.1	0.53	692.4	2.12
4.79	0.07	25.73	0.22	138.0	0.56	740.5	2.32
5.13	0.09	27.51	0.23	147.6	0.59	792.0	2.53
5.48	0.10	29.43	0.24	157.9	0.62	847.1	2.73
5.87	0.11	31.47	0.24	168.9	0.64	905.9	2.94
6.27	0.12	33.66	0.25	180.6	0.67	968.9	3.15
6.71	0.12	36.00	0.26	193.1	0.69	1036.2	3.35
7.18	0.13	38.50	0.26	206.6	0.71	1108.3	3.55
7.67	0.13	41.18	0.26	220.9	0.72	1185.3	3.72
8.21	0.14	44.04	0.27	236.3	0.74	1267.7	3.82
8.78	0.14	47.10	0.27	252.7	0.76	1355.8	3.85
9.39	0.14	50.37	0.28	270.3	0.78	1450.0	3.81
10.04	0.14	53.88	0.28	289.1	0.80	1550.8	3.72
10.74	0.15	57.62	0.29	309.1	0.83	1658.6	3.57
11.49	0.15	61.63	0.30	330.6	0.86	1773.9	3.37
12.28	0.15	65.91	0.31	353.6	0.91	1897.2	3.12
13.14	0.15	70.49	0.32	378.2	0.96	2029.1	2.83
14.05	0.16	75.39	0.34	404.5	1.03	2170.1	2.52
15.03	0.16	80.63	0.35	432.6	1.11	2320.9	2.19
16.07	0.17	86.23	0.37	462.7	1.21	2482.2	1.86
17.19	0.17	92.23	0.40	494.8	1.32	2654.8	1.50
18.38	0.18	98.64	0.42	529.2	1.45	2839.3	1.08
19.66	0.19	105.5	0.45	566.0	1.60	3036.6	0.70
21.03	0.20	112.8	0.47	605.3	1.76	3247.7	0.51
22.49		120.7		647.4		3473.5	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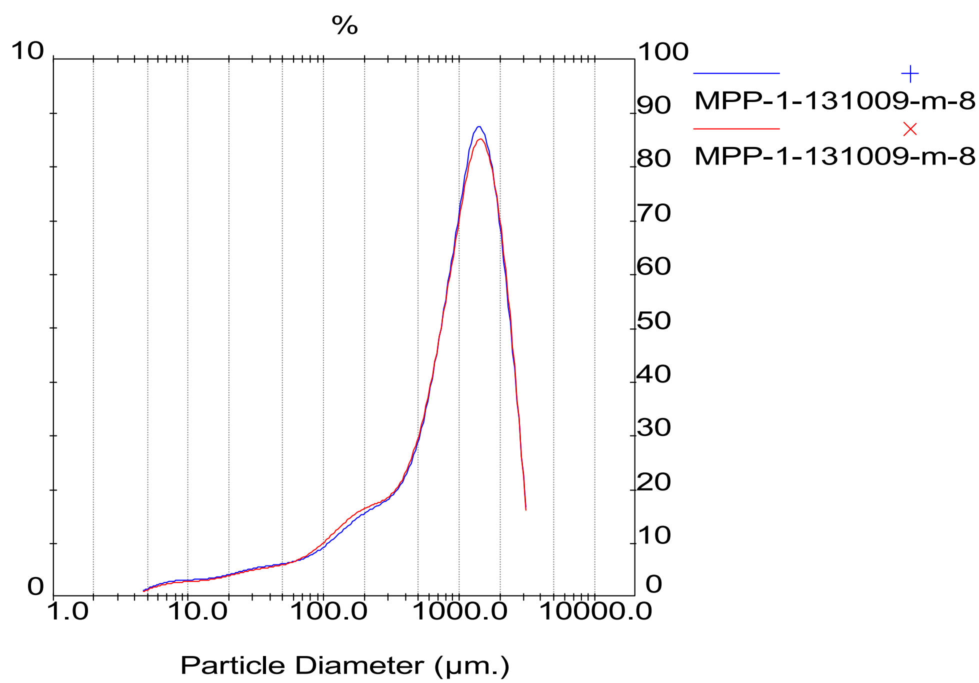
Particle Diameter (µm.)
MPP-1-131009-m-8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Particle Diameter (µm.)
MPP-1-131009-m-8
MPP-1-131009-m-8

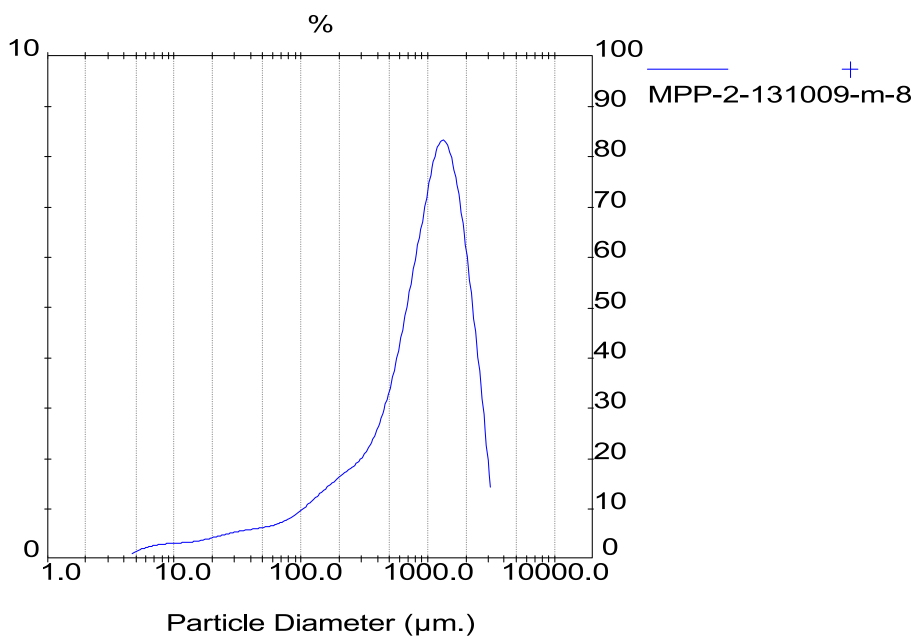
Result: Histogram Table

ID: MPP-2-131009-m	Run No: 8	Measured:
File: 2PEIRO~1	Rec. No: 41	Analysed: 14/10/2009 12:48
Path: K:\		Source: Averaged

Range: 1000 mm	Beam: 2.40 mm	Sampler:	Obs': 14.7 %
Presentation: 3OHD		Analysis: Polydisperse	Residual: 2.138 %
Modifications: None			

Conc. = 0.4522 %Vol	Density = 1.000 g/cm^3	S.S.A.= 0.0368 m^2/g
Distribution: Volume	D[4, 3] = 1039.28 um	D[3, 2] = 163.23 um
D(v, 0.1) = 105.17 um	D(v, 0.5) = 943.38 um	D(v, 0.9) = 2114.95 um
Span = 2.130E+00	Uniformity = 6.516E-01	

Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %
4.19	0.05	22.49	0.21	120.7	0.52	647.4	2.15
4.48	0.05	24.05	0.22	129.1	0.55	692.4	2.33
4.79	0.07	25.73	0.22	138.0	0.58	740.5	2.51
5.13	0.08	27.51	0.23	147.6	0.61	792.0	2.69
5.48	0.10	29.43	0.24	157.9	0.64	847.1	2.87
5.87	0.10	31.47	0.24	168.9	0.66	905.9	3.04
6.27	0.11	33.66	0.25	180.6	0.69	968.9	3.23
6.71	0.12	36.00	0.26	193.1	0.71	1036.2	3.41
7.18	0.13	38.50	0.26	206.6	0.74	1108.3	3.55
7.67	0.13	41.18	0.27	220.9	0.76	1185.3	3.63
8.21	0.14	44.04	0.27	236.3	0.79	1267.7	3.67
8.78	0.14	47.10	0.28	252.7	0.82	1355.8	3.64
9.39	0.14	50.37	0.28	270.3	0.85	1450.0	3.56
10.04	0.14	53.88	0.29	289.1	0.88	1550.8	3.43
10.74	0.14	57.62	0.30	309.1	0.92	1658.6	3.25
11.49	0.15	61.63	0.31	330.6	0.97	1773.9	3.04
12.28	0.15	65.91	0.32	353.6	1.03	1897.2	2.79
13.14	0.15	70.49	0.33	378.2	1.11	2029.1	2.52
14.05	0.16	75.39	0.35	404.5	1.19	2170.1	2.23
15.03	0.16	80.63	0.36	432.6	1.29	2320.9	1.93
16.07	0.17	86.23	0.39	462.7	1.40	2482.2	1.63
17.19	0.17	92.23	0.41	494.8	1.52	2654.8	1.31
18.38	0.18	98.64	0.43	529.2	1.66	2839.3	0.94
19.66	0.19	105.5	0.46	566.0	1.82	3036.6	0.60
21.03	0.20	112.8	0.49	605.3	1.97	3247.7	0.42
22.49		120.7		647.4		3473.5	



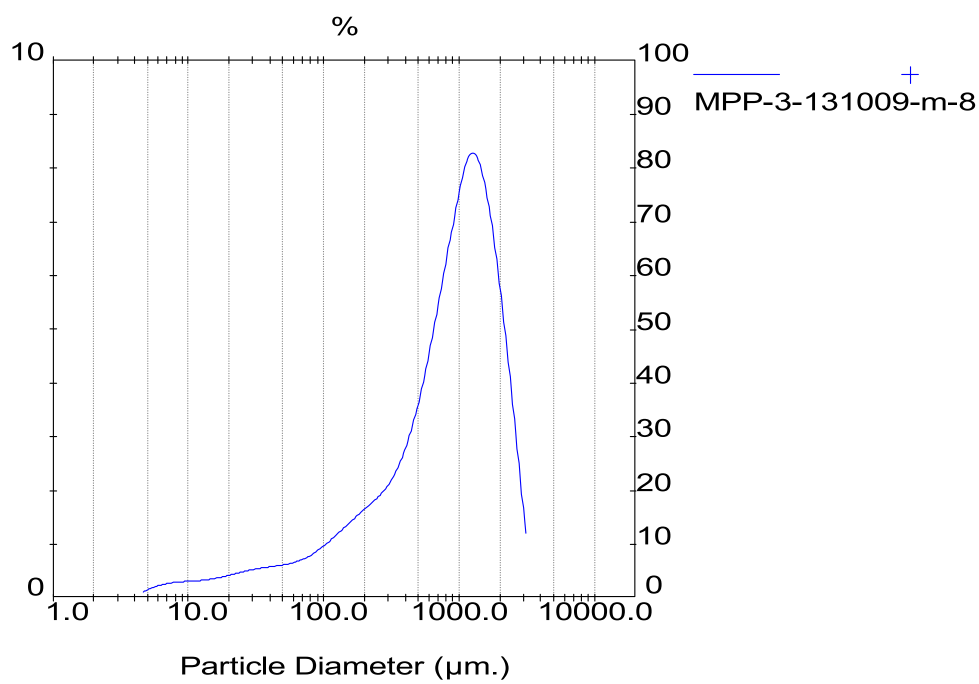
Result: Histogram Table

ID: MPP-3-131009-m	Run No: 8	Measured:
File: 2PEIRO~1	Rec. No: 46	Analysed: 14/10/2009 13:08
Path: K:\		Source: Averaged

Range: 1000 mm	Beam: 2.40 mm	Sampler:	Obs': 15.6 %
Presentation: 3OHD		Analysis: Polydisperse	Residual: 1.228 %
Modifications: None			

Conc. = 0.4501 %Vol	Density = 1.000 g/cm^3	S.S.A.= 0.0364 m^2/g
Distribution: Volume	D[4, 3] = 1008.45 um	D[3, 2] = 164.65 um
D(v, 0.1) = 107.57 um	D(v, 0.5) = 908.99 um	D(v, 0.9) = 2051.67 um
Span = 2.139E+00	Uniformity = 6.560E-01	

Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %
4.19	0.05	22.49	0.21	120.7	0.52	647.4	2.28
4.48	0.05	24.05	0.21	129.1	0.55	692.4	2.45
4.79	0.06	25.73	0.22	138.0	0.58	740.5	2.63
5.13	0.08	27.51	0.23	147.6	0.61	792.0	2.81
5.48	0.09	29.43	0.23	157.9	0.64	847.1	2.98
5.87	0.10	31.47	0.24	168.9	0.67	905.9	3.15
6.27	0.11	33.66	0.25	180.6	0.70	968.9	3.32
6.71	0.12	36.00	0.25	193.1	0.72	1036.2	3.48
7.18	0.12	38.50	0.26	206.6	0.75	1108.3	3.59
7.67	0.13	41.18	0.26	220.9	0.78	1185.3	3.64
8.21	0.13	44.04	0.26	236.3	0.81	1267.7	3.63
8.78	0.14	47.10	0.27	252.7	0.84	1355.8	3.57
9.39	0.14	50.37	0.28	270.3	0.88	1450.0	3.46
10.04	0.14	53.88	0.28	289.1	0.92	1550.8	3.31
10.74	0.14	57.62	0.29	309.1	0.97	1658.6	3.10
11.49	0.14	61.63	0.30	330.6	1.03	1773.9	2.88
12.28	0.15	65.91	0.31	353.6	1.10	1897.2	2.62
13.14	0.15	70.49	0.33	378.2	1.18	2029.1	2.34
14.05	0.15	75.39	0.34	404.5	1.28	2170.1	2.06
15.03	0.16	80.63	0.36	432.6	1.38	2320.9	1.76
16.07	0.17	86.23	0.38	462.7	1.50	2482.2	1.45
17.19	0.17	92.23	0.41	494.8	1.63	2654.8	1.14
18.38	0.18	98.64	0.43	529.2	1.78	2839.3	0.80
19.66	0.19	105.5	0.46	566.0	1.94	3036.6	0.51
21.03	0.20	112.8	0.49	605.3	2.10	3247.7	0.36
22.49		120.7		647.4		3473.5	



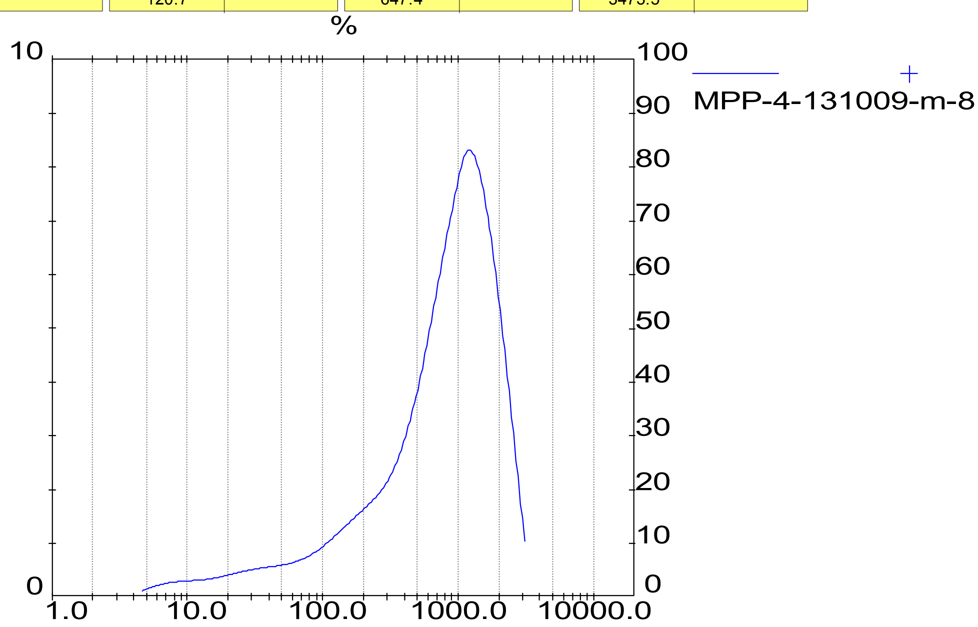
Result: Histogram Table

ID: MPP-4-131009-m	Run No: 8	Measured:
File: 2PEIRO~1	Rec. No: 51	Analysed: 14/10/2009 13:14
Path: K:\		Source: Averaged

Range: 1000 mm	Beam: 2.40 mm	Sampler:	Obs': 16.7 %
Presentation: 3OHD		Analysis: Polydisperse	Residual: 1.533 %
Modifications: None			

Conc. = 0.6032 %Vol	Density = 1.000 g/cm^3	S.S.A.= 0.0355 m^2/g
Distribution: Volume	D[4, 3] = 989.93 um	D[3, 2] = 168.86 um
D(v, 0.1) = 113.21 um	D(v, 0.5) = 889.29 um	D(v, 0.9) = 2005.84 um
Span = 2.128E+00	Uniformity = 6.515E-01	

Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %
4.19	0.04	22.49	0.20	120.7	0.51	647.4	2.40
4.48	0.05	24.05	0.21	129.1	0.54	692.4	2.58
4.79	0.06	25.73	0.21	138.0	0.57	740.5	2.75
5.13	0.08	27.51	0.22	147.6	0.60	792.0	2.93
5.48	0.09	29.43	0.23	157.9	0.63	847.1	3.09
5.87	0.10	31.47	0.23	168.9	0.66	905.9	3.25
6.27	0.11	33.66	0.24	180.6	0.69	968.9	3.42
6.71	0.11	36.00	0.24	193.1	0.72	1036.2	3.56
7.18	0.12	38.50	0.25	206.6	0.75	1108.3	3.65
7.67	0.12	41.18	0.25	220.9	0.78	1185.3	3.66
8.21	0.13	44.04	0.25	236.3	0.81	1267.7	3.62
8.78	0.13	47.10	0.26	252.7	0.85	1355.8	3.52
9.39	0.13	50.37	0.27	270.3	0.89	1450.0	3.39
10.04	0.13	53.88	0.27	289.1	0.94	1550.8	3.21
10.74	0.14	57.62	0.28	309.1	1.00	1658.6	3.00
11.49	0.14	61.63	0.29	330.6	1.07	1773.9	2.76
12.28	0.14	65.91	0.30	353.6	1.15	1897.2	2.50
13.14	0.14	70.49	0.32	378.2	1.24	2029.1	2.22
14.05	0.15	75.39	0.33	404.5	1.34	2170.1	1.94
15.03	0.15	80.63	0.35	432.6	1.46	2320.9	1.64
16.07	0.16	86.23	0.37	462.7	1.59	2482.2	1.33
17.19	0.17	92.23	0.39	494.8	1.73	2654.8	1.03
18.38	0.17	98.64	0.42	529.2	1.89	2839.3	0.70
19.66	0.18	105.5	0.45	566.0	2.05	3036.6	0.44
21.03	0.19	112.8	0.48	605.3	2.22	3247.7	0.30
22.49		120.7		647.4		3473.5	



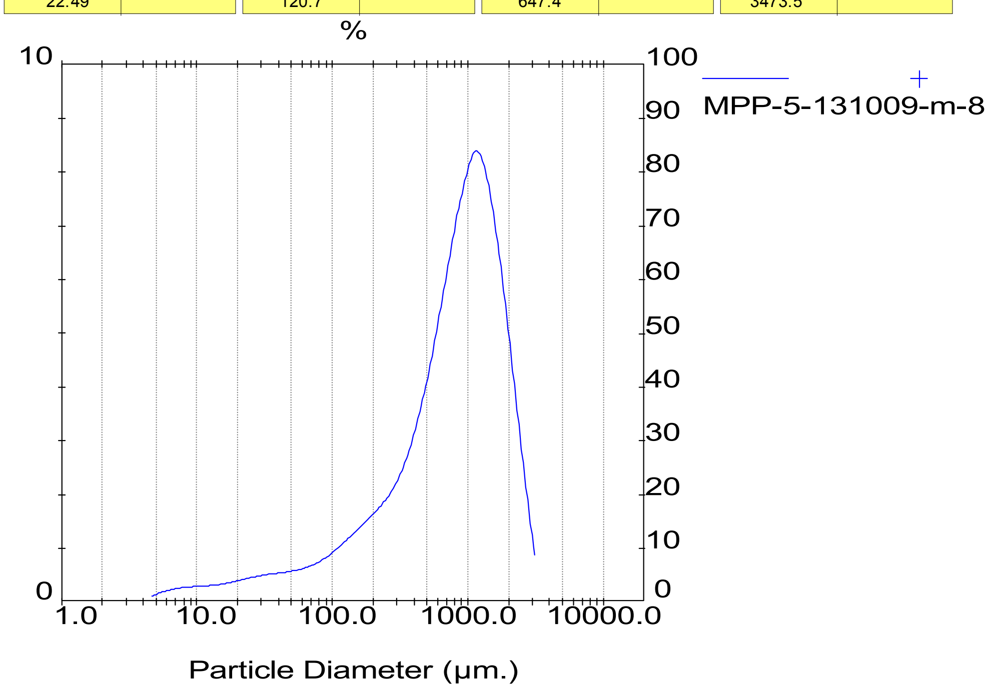
Result: Histogram Table

ID: MPP-5-131009-m | Run No: 8 | Measured:
File: 2PEIRO~1 | Rec. No: 56 | Analysed: 14/10/2009 13:26
Path: K:\ | Source: Averaged

Range: 1000 mm | Beam: 2.40 mm | Sampler: | Obs': 17.8 %
Presentation: 3OHD | Analysis: Polydisperse | Residual: 1.425 %
Modifications: None

Conc. = 0.7340 %Vol | Density = 1.000 g/cm^3 | S.S.A.= 0.0349 m^2/g
Distribution: Volume | D[4, 3] = 957.22 um | D[3, 2] = 172.04 um
D(v, 0.1) = 118.21 um | D(v, 0.5) = 856.55 um | D(v, 0.9) = 1926.44 um
Span = 2.111E+00 | Uniformity = 6.462E-01

Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %
4.19	0.04	22.49	0.19	120.7	0.50	647.4	2.57
4.48	0.05	24.05	0.20	129.1	0.53	692.4	2.75
4.79	0.06	25.73	0.21	138.0	0.56	740.5	2.93
5.13	0.08	27.51	0.21	147.6	0.59	792.0	3.11
5.48	0.09	29.43	0.22	157.9	0.62	847.1	3.27
5.87	0.09	31.47	0.22	168.9	0.65	905.9	3.41
6.27	0.10	33.66	0.23	180.6	0.69	968.9	3.55
6.71	0.11	36.00	0.23	193.1	0.72	1036.2	3.65
7.18	0.12	38.50	0.24	206.6	0.75	1108.3	3.70
7.67	0.12	41.18	0.24	220.9	0.79	1185.3	3.67
8.21	0.12	44.04	0.25	236.3	0.83	1267.7	3.59
8.78	0.13	47.10	0.25	252.7	0.87	1355.8	3.45
9.39	0.13	50.37	0.26	270.3	0.92	1450.0	3.27
10.04	0.13	53.88	0.26	289.1	0.98	1550.8	3.06
10.74	0.13	57.62	0.27	309.1	1.05	1658.6	2.82
11.49	0.13	61.63	0.28	330.6	1.13	1773.9	2.56
12.28	0.14	65.91	0.29	353.6	1.22	1897.2	2.27
13.14	0.14	70.49	0.31	378.2	1.33	2029.1	1.98
14.05	0.14	75.39	0.32	404.5	1.44	2170.1	1.69
15.03	0.15	80.63	0.34	432.6	1.57	2320.9	1.40
16.07	0.16	86.23	0.36	462.7	1.72	2482.2	1.13
17.19	0.16	92.23	0.39	494.8	1.87	2654.8	0.86
18.38	0.17	98.64	0.41	529.2	2.04	2839.3	0.59
19.66	0.18	105.5	0.44	566.0	2.21	3036.6	0.37
21.03	0.18	112.8	0.47	605.3	2.38	3247.7	0.25
22.49		120.7		647.4		3473.5	



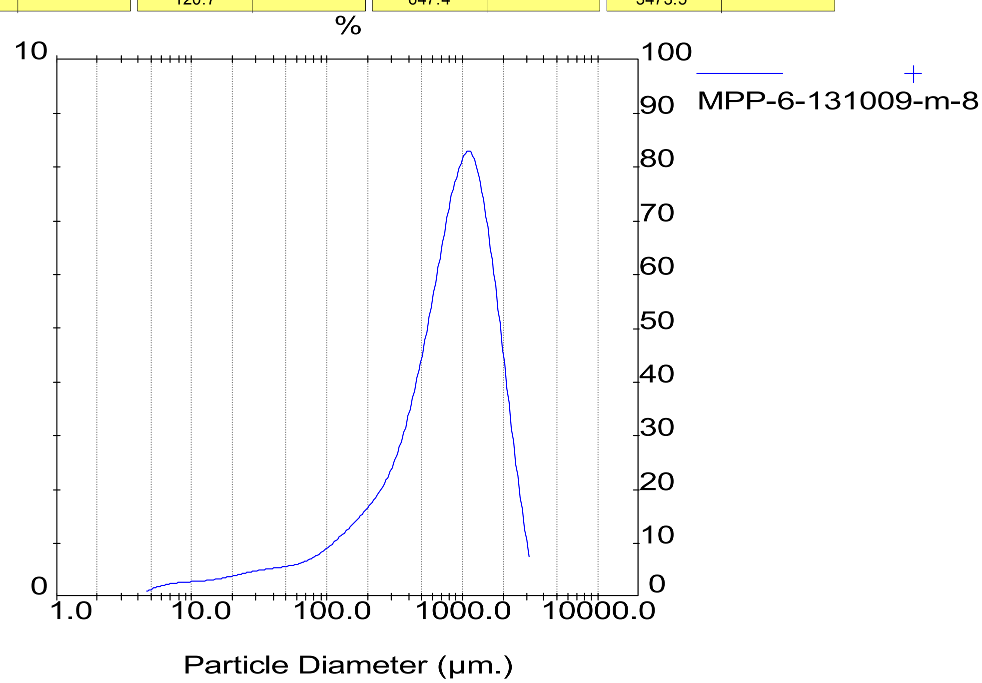
Result: Histogram Table

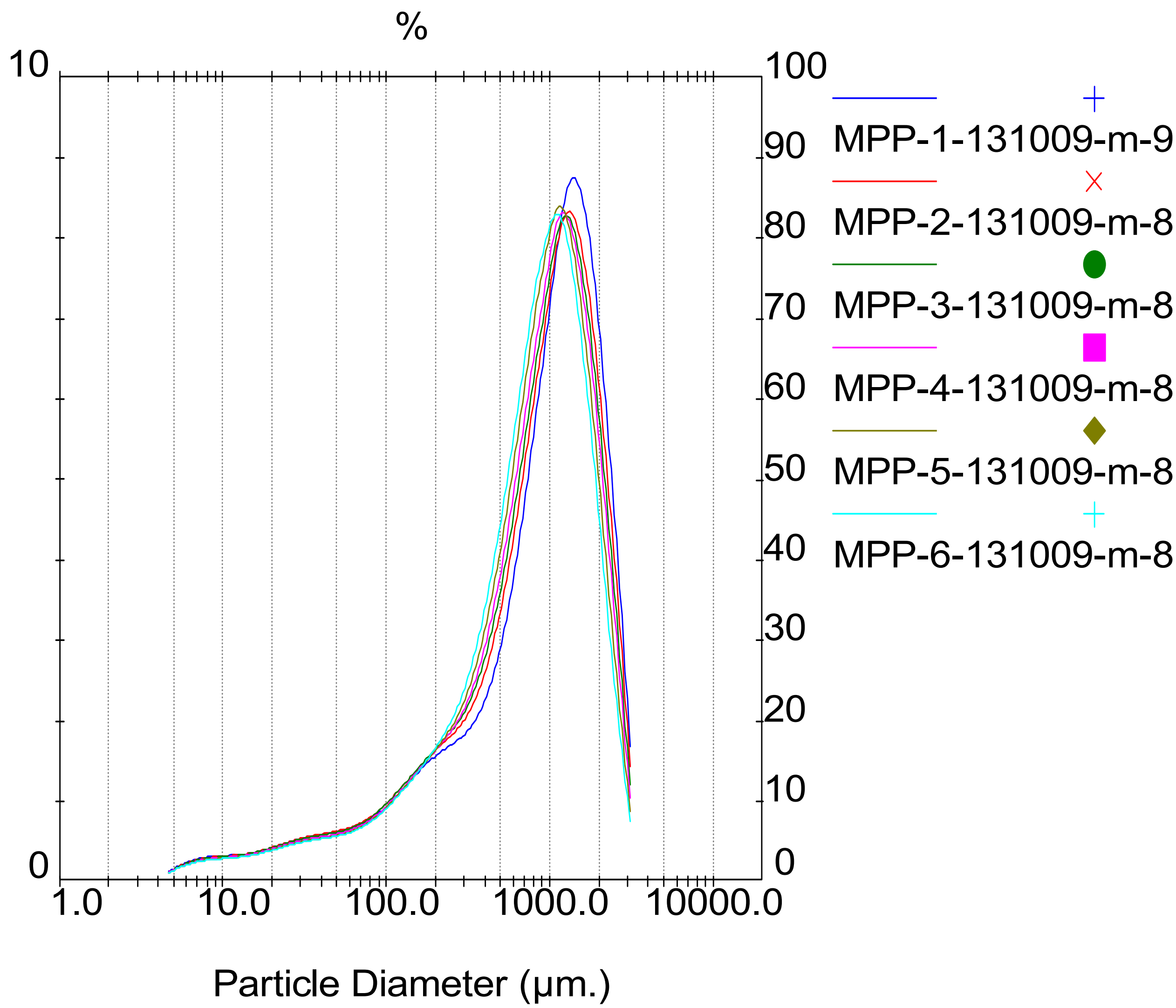
ID: MPP-6-131009-m	Run No: 8	Measured:
File: 2PEIRO~1	Rec. No: 61	Analysed: 14/10/2009 13:35
Path: K:\		Source: Averaged

Range: 1000 mm	Beam: 2.40 mm	Sampler:	Obs': 17.4 %
Presentation: 3OHD		Analysis: Polydisperse	Residual: 1.396 %
Modifications: None			

Conc. = 0.3343 %Vol	Density = 1.000 g/cm^3	S.S.A.= 0.0348 m^2/g
Distribution: Volume	D[4, 3] = 924.61 um	D[3, 2] = 172.32 um
D(v, 0.1) = 119.83 um	D(v, 0.5) = 820.28 um	D(v, 0.9) = 1860.67 um
Span = 2.122E+00	Uniformity = 6.479E-01	

Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %	Size (um)	Volume In %
4.19	0.04	22.49	0.19	120.7	0.49	647.4	2.72
4.48	0.05	24.05	0.20	129.1	0.52	692.4	2.90
4.79	0.06	25.73	0.20	138.0	0.56	740.5	3.08
5.13	0.07	27.51	0.21	147.6	0.59	792.0	3.25
5.48	0.09	29.43	0.22	157.9	0.62	847.1	3.38
5.87	0.09	31.47	0.22	168.9	0.66	905.9	3.49
6.27	0.10	33.66	0.23	180.6	0.69	968.9	3.58
6.71	0.11	36.00	0.23	193.1	0.73	1036.2	3.65
7.18	0.11	38.50	0.24	206.6	0.77	1108.3	3.65
7.67	0.12	41.18	0.24	220.9	0.81	1185.3	3.58
8.21	0.12	44.04	0.24	236.3	0.86	1267.7	3.47
8.78	0.12	47.10	0.25	252.7	0.92	1355.8	3.30
9.39	0.13	50.37	0.25	270.3	0.98	1450.0	3.11
10.04	0.13	53.88	0.26	289.1	1.05	1550.8	2.88
10.74	0.13	57.62	0.27	309.1	1.13	1658.6	2.63
11.49	0.13	61.63	0.28	330.6	1.22	1773.9	2.36
12.28	0.14	65.91	0.29	353.6	1.32	1897.2	2.08
13.14	0.14	70.49	0.30	378.2	1.44	2029.1	1.78
14.05	0.14	75.39	0.32	404.5	1.57	2170.1	1.50
15.03	0.15	80.63	0.34	432.6	1.71	2320.9	1.22
16.07	0.15	86.23	0.36	462.7	1.86	2482.2	0.98
17.19	0.16	92.23	0.38	494.8	2.02	2654.8	0.75
18.38	0.17	98.64	0.41	529.2	2.19	2839.3	0.51
19.66	0.18	105.5	0.44	566.0	2.36	3036.6	0.32
21.03	0.18	112.8	0.46	605.3	2.53	3247.7	0.22
22.49		120.7		647.4		3473.5	




%
10
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10000.0
Particle Diameter (µm.)
MPP-1-131009-m-9
MPP-2-131009-m-8
MPP-3-131009-m-8
MPP-4-131009-m-8
MPP-5-131009-m-8
MPP-6-131009-m-8

8. COMMENTS

Waste Preparation Unit, concept and trade-off

Comments

Detailed comments

Page/paragraph	Comment
5/Section 1.2, first bullet	This tasks (Description of the technical specifications (RD2)) belongs to a previous phase ; RD2 is an input to the current work (see objectives) OK, removed from the description of tasks
11/Section 4.2, fifth paragraph	We found only one reference, in T71.9.4 (page 64) referred to sludge characterization during the FU optimisation tests of the prototype, but these doesn´t correspond to the Feed but to the reactor broth You are right, there is no other reference and this one has a rather indirect applicability
25/Table 6	I anticipate columns 6 and 7 are at speed 1 and columns 8 and 9 are at speed 2? In fact, during the 10 min test with faeces the first 5 min the speed was 3000 rpm and in the last 5 min it was 4000; anyway, the increase of speed didn't seem to be clearly effective
27/Section 4.6, first paragraph	Can you precise what is the compositon of the mixture you are using?same as page 20 with the Fristam pump? OK, included in the text: "in both cases performed as well with the vegetable mixture in the concentrations foreseen for the WPU nominal operation conditions"
40/Section 7, last paragraph	This paragraph maybe not clear enough: the idea would be that we're not sure that a threshold value is enough, we obtain like 90% of particles lower than …, that is another thing Included in the text: "the existing specifications needing to be revisited" *taking into account not only the specified threshold sizes for straw and vegetables (0,2 and 2 mm respectively) but as well*

	potential acceptance criteria based on particle size distribution or % of compliant volume of particles, coherent with the needs of CI compartment (Understood and agreed)