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Representative Sampling of Suspended Particulate Materials in Horizontal Ducted Flow: Evaluating the Prototype 'EF-sampler'

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Abstract

The 'EF-sampler' is a newly developed sampler for suspended particulate materials in horizontal pneumatic conveying systems, designed for maximum possible compliance with the Theory of Sampling (TOS). Hitherto no sampler for this deployment location exists on the market that ensures representative samples as defined by TOS. Because of confinement of the pressurised ducted flow and because of gravitative and flow segregation, unbiased sampling constitutes a serious challenge. In addition to the primary demand for representativeness, interference with the material flow needs to be minimized in order to prevent clogging effects and/or possible pressure surges. We here disclose all design principles of the 'EF-sampler' and validate a 1/3-scale prototype in a pneumatic test facility by presenting our first test campaign results. Testing focuses on assessing sampling representativeness (accuracy and precision) using wheat flour and pulverized alumina as the major test materials, both spiked with LDPE plastic pellets in the role as trace constituents, extraneous material or contaminants. Input pellet concentration levels served as nominal reference values for the accuracy evaluation. Test parameters include airflow rate, sample LDPE pellet concentration and different cross-cutting sampler velocities. Results show that the patented EF-sampler prototype enables to extract fit-for-purpose samples with a relative inaccuracy <5% for the stated test materials, which is highly acceptable for this most-difficult deployment context. Sampler velocity and especially the material flow dilution status impact the accuracy of sample extraction, while precision remains constantly good for all test conditions. The prototype EF-sampler is not a universal sampler, since it is designed to require situation-dependant adjustments based on specific material heterogeneity and flow regime characteristics. However, the first test campaign results on two widely different materials show conclusively that it accommodates a wide field of potential applicability for many similar types of materials.

Keywords: Horizontal sampler; Pressurised ducted flow; Representative sampling; Theory of sampling (TOS); Pneumatic conveying; Suspended particulate materials

Introduction

Pneumatic conveying systems are widely used in processing industries, i.e. systems that transport suspended aggregates/particulate matter forced by an air or gas stream through horizontal and vertical ducts. The advantages of pneumatic transportation compared to alternative mechanical conveying systems, e.g. conveyer belts, are potential economic benefits and higher flexibility in terms of rerouting or expansion, and especially complete enclosure of the material of interest. This is particularly important when dealing with pulverized materials in order to prevent material loss. Furthermore changes of the ducted material, e.g. moisture level, can be minimized by confinement.

In many cases it is important to have exact knowledge of the material properties of the material during pneumatic conveying. Reliable quality assurance of the transported material therefore makes it a requirement to be able to extract representative samples as defined by the Theory of Sampling (TOS). Pierre Gy's TOS Sampling Theory and Practice (STP) is the only comprehensive framework that allows a profound analysis of all sampling equipment, methods and procedures; this has therefore been used as the backbone for design, development, implementation and evaluation of the present sampler.

Pneumatic conveying systems are challenging, since the pressurized system cannot be arbitrarily intersected for sample extracting due to the risk of pressure loss or external discharge of material. Furthermore the sampling operation must not constrict the material flow in order to minimize the risk of clogging and/or pressure surges. Another important adverse factor is the fact that ducted horizontal flow causes significant vertical and sometimes also radial flow segregation. The prime objective in order to gain a representative sample is to overcome these adverse effects by an appropriate designing for a sampler for this specific process deployment.

In this study we describe a newly developed sampler for horizontal pneumatic conveying system, the "EF-sampler", which is in full compliance with the stringent principles laid down by TOS as possible.

A brief introduction of the most important aspects of TOS relevant for the design is given in the next paragraph, followed by a detailed description of the resulting design principles. This is followed by a discussion on the experimental test design used to validate the sampler while the main results are presented and discussed in last few paragraphs.

Representative Sampling–The Theory of Sampling (TOS)

Almost every measurement involves the process of taking samples. It is often questionable, indeed often undocumented if the extracted

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samples are truly representative, or whether samples are in reality just 'specimens', which is a TOS' term for a non-representative sub-part of a lot; such specimens are uninteresting lot extractions.

All naturally occurring materials are heterogeneous, caused by compositional differences as well as grouping and segregation of the material in the lot. The heterogeneity phenomenon makes sampling far from trivial and requires solid knowledge about heterogeneity, and especially how to counteract its effects in the sampling process. Since more than 60 years, Pierre Gy's Theory of Sampling (TOS) has reigned as the only complete theoretical and practical framework for representative sampling. In particular, TOS shows how to sample in an unbiased fashion ("correct sampling") which is the prerequisite for representative sampling from all sorts of materials and lot types. TOS' principles demand an equal probability of increments of the lot to be sampled, which is the critical guarantee for non-biased sampling. TOS' derived Sampling Theory and Practice (STP) furthermore enables to analyse sampling methods as well as sampling procedures and equipment types with respect to the principle of representativeness. Non-representative samples are primarily caused by so-called 'incorrect sampling errors', which must be identified and eliminated, or at least be reduced significantly. Such sampling process errors will unavoidably lead to an inconstant sampling-bias, which cannot be corrected for, leading to uncontrolled inflation of the total sampling error. For a complete introduction to TOS the reader is referred to the following selected literature [1-7]. The main definitions of TOS, as used in the following, are provided in the following (Table 1).

Terms of TOS	Definition
Sample	Correctly extracted material from the lot, which only originates from a qualified sampling process ("sampling correctness").
Composite sample	Aggregation of several increments – a composite sample constitutes "physical averaging".
Specimen	A 'sample' that cannot be documented to be representative.
Increment	Correctly delineated, materialised sampling units of the lot. Composite samples result from an increment aggregation process.
Fragment	Smallest separable unit of the lot, e.g. mineral grain, kernel, biological cell etc. that is not affected by the sampling process itself. By naming the smallest unit-of-interest a fragment, TOS allows to treat even the situation in which the sampling process results in fragmentation of (some) of the original units.
Lot	Sampling target, e.g. truck load, railroad car, ship's cargo, batch etc. Lot refers both to the physical, geometrical form as well as the physic-chemical characteristics of the material being subject to sampling. Lots can be either stationary or dynamic (moving).
Lot dimensionality	TOS distinguishes between 0-, 1-, 2- and 3-dimensional lots. A 0-dimensional lot can be manipulated (forcefully mixed, moved etc.) in its entirety without undue efforts.
Scale	Heterogeneity, and counter-acting sampling efficiency, is influential at all scales from increment to lot. Correct sampling is scale-invariant, i.e. the same principles apply to all relevant scales in the sampling pathway.
Heterogeneity	Heterogeneity is the prime characterisation of all naturally occurring materials, including industrial lots. Heterogeneity manifests itself at all scales related to sampling for nearly all lot and material types. The only exception is uniform materials[1], which however are such rare occurrences that no generalisation w.r.t. general sampling can be made here from.
Sampling correctness	Elimination of sampling bias, by correct design, performance and maintenance of the sampling process/equipment. In the event of sampling correctness, only sampling precision remains, which is a much easier issue to control for within specified limits.
Representativeness	Representativeness implies both correctness as well as a sufficiently small sampling reproducibility (sampling precision).

Table 1: Fundamental concepts and definitions in Theory of Sampling. See also literature cited immediately above for a more fully developed introduction to TOS and TSP.

[1]Uniform materials: Materials with a repeated (correct) sampling reproducibility lower than 2% (or lower still, various definitions pertain to different sciences and technology fields). Such materials do only very rarely occur naturally however (exception gasses and infinitely diluted solutions etc.).

In the development phase of any new sampler, preventing incorrect sampling errors by correct design principles must have the absolutely highest priority. Unfortunately not all OEMs of sampling equipment respect these principles, and thus do not necessarily manufacture bias-generating sampling equipment, a situation which is also partly caused by misleading and/or incomplete sampling standards [8]. For a better understanding of the design principles of the presented horizontal sampler, a brief of the main principles of TOS is given below.

The critical criterion for representativeness is expressed by the 'Fundamental Sampling Principle (FSP)', stating that all fragments (grains, particles) in the lot must have the same none-zero probability of ending up in the final sample [1]. This implies that elements not belonging to the material lot must have a zero probability of being extracted. FSP applies identically at higher scale dimensions, in particular governing extraction of increments, which are the lot volume elements of paramount interest for practical sampling. The final sample, termed a composite sample, should consist of an appropriate number of increments, which is related to the effective heterogeneity of the lot material. It is an often disregarded or fully unknown fact that the number of increments needed to counteract a specific material heterogeneity is not related to the size (mass) of the lot, but solely to the magnitude of the heterogeneity (sic). A recent comprehensive illustration of this issue can be found in [9-12].

In contrast to unitary sampling operations ('grab sampling'), which disobey the FSP and therefore never can achieve sampling correctness and therefore neither representativeness, composite sampling actively counteracts the inherent lot heterogeneity.

In contrast to many sampling standards and guides specifying measurement uncertainty, TOS defines the term 'representative' explicitly with full theoretical and practical rigour. According to TOS, sampling processes can be considered as representative if, and only if, samples are extracted by procedures, which are both 'accurate' and 'precise' [1]. Accuracy of a sampling process is achieved when the average sampling error equals zero or is effectively confined to be below a predetermined acceptable low value; otherwise the sampling process is biased. Likewise a sampling process can be rated as precise if the variance of the sampling error is below a predetermined acceptable value. Mathematical expressions of the defined terms can be found in the TOS literature.

The representativeness of a sampling process can be compromised by the effects of several types of sampling errors. In TOS these error sources are subdivided in 'incorrect sampling errors' and 'correct sampling errors' (and two, much more easily handled process sampling errors). The perhaps paradoxical term, 'correct sampling errors' expresses the situation that these errors occur even when the sampling process itself is 'correct'. The following figure gives a schematic

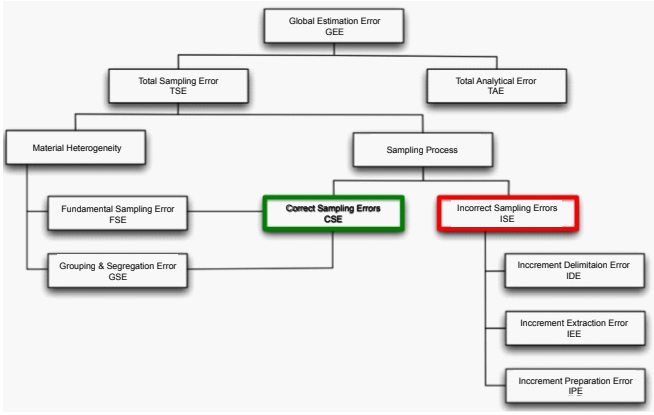

Figure 1: Relation of TOS' basic five sampling errors in stationary and process sampling situations, highlighting *correct* and *incorrect* sampling errors. Source: Wagner, Esbensen 2012.

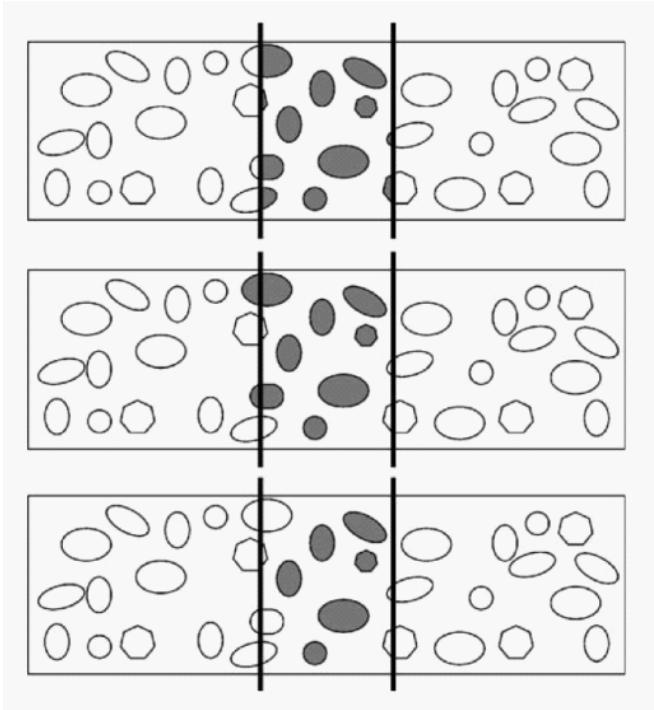

Figure 2: Illustration of the centre-of-gravity rule with grey shadings demonstrating material, which must end up in the final sample, lest IEE occur. Upper figure: correct theoretical extraction. Center: correct practical extraction. Bottom picture: *incorrect* extraction, disobeying the centre-of-gravity rule. Source: Gy, 1993, Pitard, 1993; Smith, 2001; Petersen et al., 2004.

overview of TOS' classification of the basic five sampling errors for stationary lot sampling. These are also the five major influencing errors occurring in process sampling.

The sum of the effects of all error sources is termed 'Global Estimation Error' (GEE), consisting of the 'Total Sampling Error' (TSE) and the 'Total Analytical Error' (TAE). Uncertainties of analytical results are expressed as the variance of the TAE, while all other error sources are summed up under the term 'Total Sampling Error'. It is essential to notice that for significantly heterogeneous materials TAE is nearly always much smaller than the sum of all sampling errors (up to 10-100 times smaller), while many guidelines and even some standards dealing with "uncertainty estimation" still entirely focus on TAE as the only error source [7].

The schematic overview in (Figure 1) shows that both the material heterogeneity and the sampling process can cause these types of sampling errors to occur. The 'Correct Sampling Errors' (CSE) comprise the Fundamental Sampling Error (FSE) and the Grouping & Segregation Error (GSE), both caused by material heterogeneity. TOS differentiates between constitutional heterogeneity (CH) and distributional heterogeneity (DH). CH represents the component of heterogeneity, which depends on the physical and/or chemical differences between individual fragments in the lot material, causing the Fundamental Sampling Error. The constitutional heterogeneity increases when the compositional difference between fragments increases. FSE can only be reduced (but never completely eliminated)

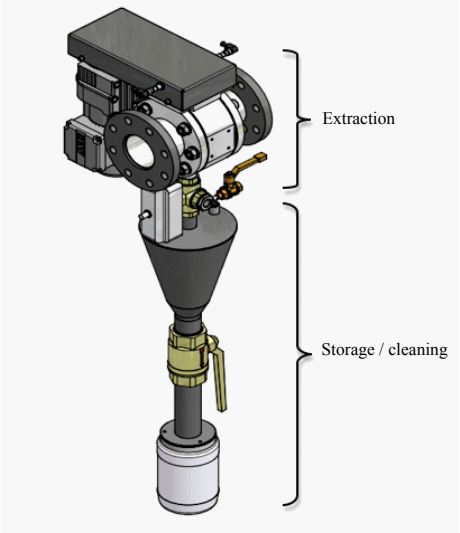

Figure 3: Schematic overview of EF-sampler. Upper part showing extraction mechanism including electric power supply and extraction mechanism with enclosed sampling arm, lower part represents the storage/cleaning section including compositing cylinder, pressure valve, storage valve and storage container.

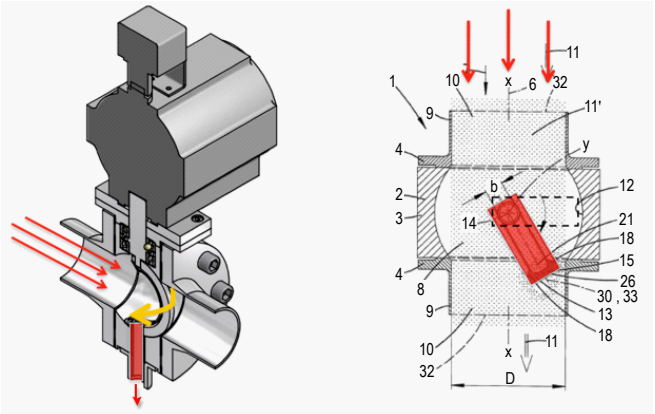

Figure 4: Schematic overview of the EF-sampler. Left figure showing side view illustrating material flow direction, rotational movement of cutter arm and recovery of extracted material through outlet chute. Right figure shows top view of sampler, highlighting the rotational movement of the sampling arm, ducted material flow direction (top arrows) and parking position of the sampling arm (dashed line).

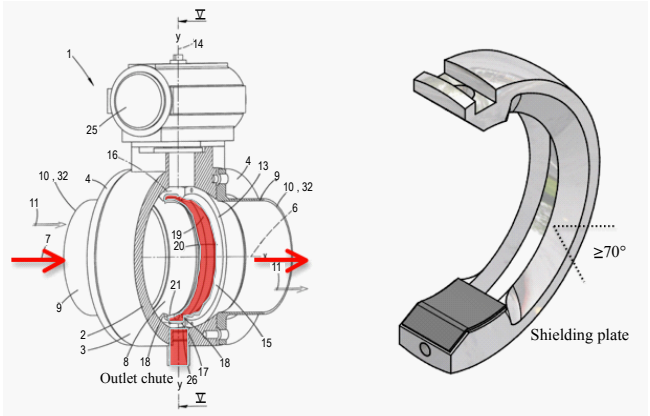

Figure 5: Side view of EF-sampler (left figure), highlighting cutter arm, material flow direction and outlet chute for material extraction. Right figure depicts details of the replacable sampling arm with inclination of cutter blade edges and shielding plate for the extraction opening.

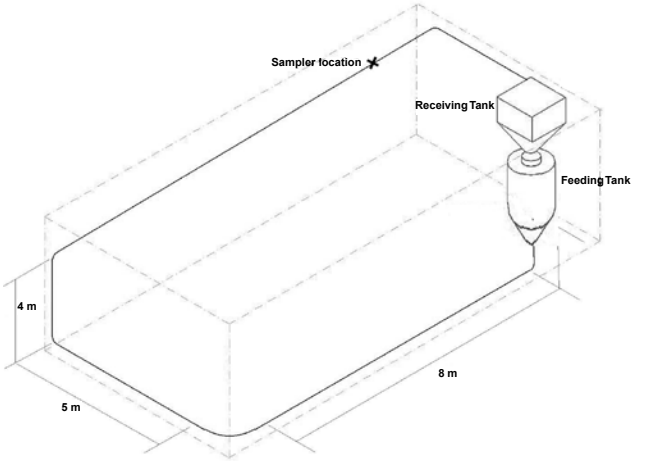

Figure 6: Schematic drawing of pneumatic transportation system (ca. 30m) including sampler location (marked with 'X'), receiving tank and feeding tank (volume ca. 1,5m3). Rotational feeder located below feeding tank is not shown. Source: POSTEC (modified by the current authors).

by comminution (crushing), followed by mixing, meaning that FSE is always present to a certain degree [1]. The second correct sampling error is termed 'Grouping & Segregation Error' (GSE) and is caused by the distributional heterogeneity, meaning the inherent tendency of particles to group and segregate at scales commensurate with the increment volume and upwards only limited by the size of the entire lot. Since it is the distributional heterogeneity, which basically is also dependent on the spatial distribution of all individual fragments or groups of fragments (i.e. increments) in the lot, that causes the GSE, an effective counteraction process for minimizing this error source is either mixing before sampling and/or using a higher number of smaller increment volume, together better "covering" the lot volume [13,6,7].

Contrasting FSE and GSE, the incorrect sampling errors can, and must by all means be minimized, indeed preferentially be eliminated from the sampling process - of course this is an issue of foremost interest in the design phase of a new sampler. These three bias-generating errors (IDE- Increment Delimitation Error, IEE-Increment Extraction Error and IPE- Increment Preparation Error) cannot be corrected for by any a posteori procedures, statistics or data analysis, since a sampling bias is never constant and therefore cannot subject to the standard statistical "bias correction".

The 'Increment Delimitation Error' (IDE) can occur in connection with delineating the increments for physical extraction. In order to ensure that the Fundamental Sampling Principle is obeyed (equal likelihood for all fragments in the lot of ending up in the sample), IDE can only be avoided by ensuring that the geometrical delineation of the increment completely covers the relevant dimensions of the lot. In TOS lot dimensionality is not only related to the physical geometry of the lot only (e.g. material on conveyer belts or in pipes, stockpiles etc.) but also refers to the operative number of dimension that are 'covered' during the sampling process. Thus TOS considers 1-, 2-, and 3-dimensional sampling lots, whereas the special case of 0-dimensional refers to a lot that can be effectively manipulated: moved, mixed and sampled with complete correctness. Ideally every 2-D and 3-D lot should be transformed to a 1-dimensional sampling situation [2,1,7], i.e. into a sampling situation for which one dimension in space dominates (e.g. process streams, pipelines, conveyer belts etc.). This configuration, i.e. process sampling facilitates correct delineation of increments consisting of the material from the complete depth and width of the source stream, effectively reducing the lot heterogeneity to one dimension-the longitudinal dimension of the material flow direction. Horizontal pipe sections, as in the present context of the new EF-sampler, constitute typical one-dimensional lot examples. A correct delineation of increments can only be achieved by extracting a complete cross-sectional slice of the material stream with constant width, fully reproducible over time. A prerequisite is that the cutting planes define the increment sides, must be strictly parallel, perpendicular to the material flow.

The second incorrect sampling error is termed 'Increment Extraction Error' (IEE), occurs if/when particles inside the delineated increment in fact do not end up in the final sample. This is also referred to as the 'centre-of-gravity rule' in TOS stating that particles/fragments, which have their centre of gravity inside the delineated increment, must end up in the final sample [1,2]. (Figure 2) illustrates this rule; grey shading represents material, which must end up in the final samples, lest an IEE occurs. The upper figure shows the correct theoretical extraction, whereas the centre figure shows the correct

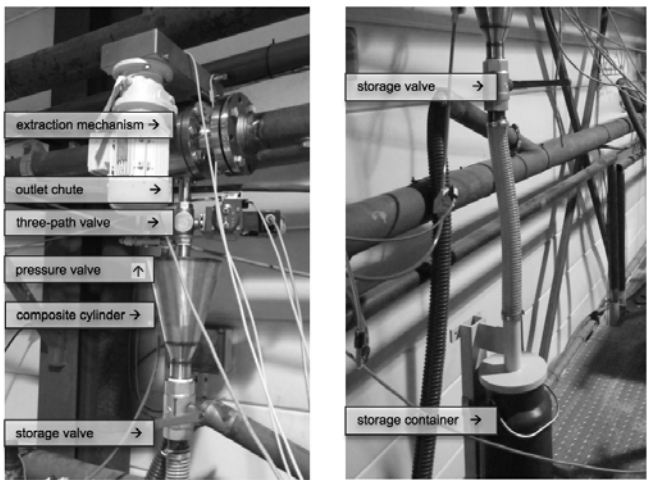

Figure 7: E-F-sampler installed at test facility. Extraction and upper storage section (left) including electric power supply, extraction mechanism and compositing cylinder. Right: close-up of lower storage section showing storage valve and storage container.

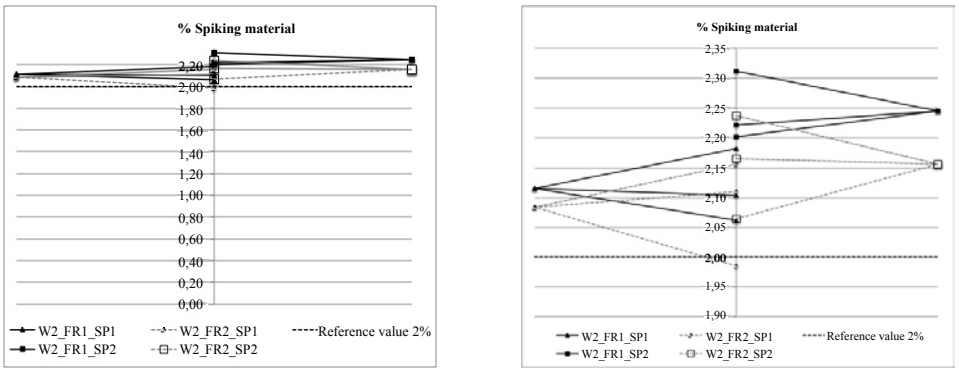

Figure 8: Sampling results of wheat flour with 2% spiking concentration, comparing SP1, SP2, FR1 and FR2 – 3 repetition rounds (left). Close-up of variations of sampling results (right). Horizontal stippled line represents the nominal reference value.

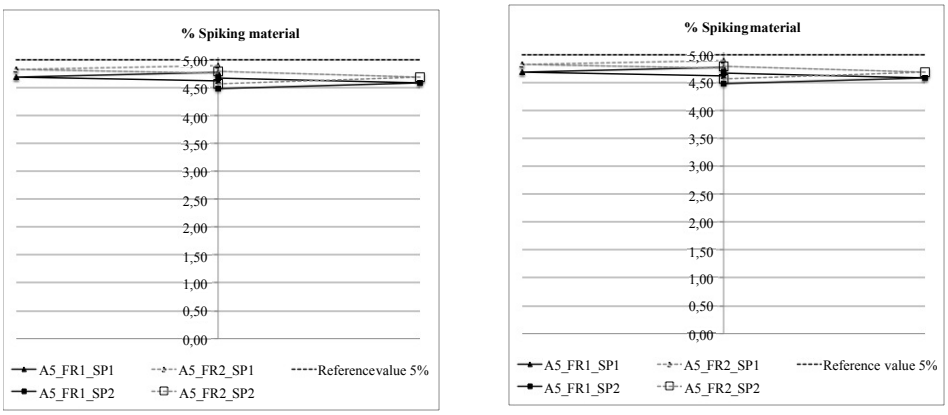

Figure 9: Sampling results of alumina with 5% spiking concentration, comparing SP1, SP2, FR1 and FR2 – 2 repetition rounds (left). Close-up of variations of sampling results (right). Horizontal stippled line represents the nominal reference value.

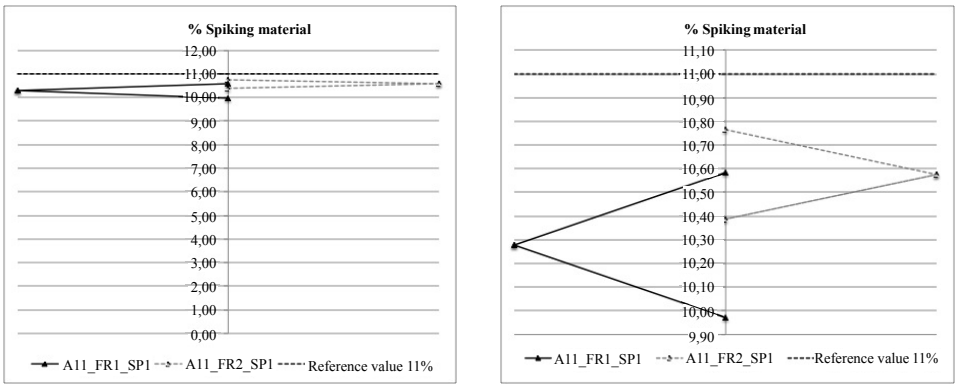

Figure 10: Sampling results of alumina with 11% spiking concentration, comparing SP1, FR1 and FR2 – 2 repetition rounds (left). Close-up of variations of sampling results (right). Horizontal stippled line represents the nominal reference value.

practical extraction. The bottom figure gives an example of incorrect extraction, disobeying the centre-of-gravity rule and therefore causing an IEE. Sufficient depth and volume of the sampler, correct inclination of cutter blade sides for preventing particles from climbing up the edges into the sample volume, as well as a limited, predefined cross-cutting velocity are factors that effectively can prevent IEE and which must therefore be carefully considered in the sampler design phase. Examples of the effects causing IEEs can be

1) Particles bouncing off the sampling tool edges

2) Particles ending up in the final sample, which do not belong to the delineated increment or

3) Fine particles that are blown away before extraction. The latter requires that the sampling extraction mechanism (e.g. cutter) is fully closed preventing loss of material. Since the material, which is interacting with the extraction cutter is of varying composition and

disposition (lot heterogeneity); IEE is an intermittent, irregularly varying error.

The third IEE is termed 'Increment Preparation Error' (IPE). IPE may occur when samples or increments are modified, in whatever fashion, after extraction, including alterations like evaporation, moisture absorption, loss of material as well as deliberate manipulation as fraud or sabotage. To limit the occurrence of a potential IPE appropriate (correct) sample handling and laboratory protocols are imperative, regulating i.e. sealing, storage and documentation of final samples. IPE is a type of sampling error, which can be brought under complete control, only depending on knowledge, willingness and diligence.

Pitard [2] contains a comprehensive, and perfunctory, survey and many in-depth examples of the issues involved regarding all ISE and the critical ways and means to counteract their effects for both stationary as well as dynamic lots.

In many cases elimination of incorrect sampling errors demands only minor but decisive changes in the sampling process, while other incorrect sampling errors are caused by a faulty design of the sampler or the sampling system itself. This study also serves the purpose to point out the importance of integrating TOS in the design phase of new samplers and shows an example of how this can be achieved for a very challenging deployment scenario.

Design Principles of EF-Sampler

The new horizontal sampler, the 'E-F-sampler', is a fully operational 1/3-scale version constructed to fit into horizontal pipe sections with a diameter of 76 mm (3-inches). The fundamental design principles of the sampling mechanism of a preliminary prototype were presented and preliminary discussed at the 5th World Conference on Sampling and Blending [14]. In the present work all design principles of the final scale-model sampler version are disclosed, which have now been verified in a dedicated test campaign.

Even though many industries use horizontal pneumatic transportation systems, no satisfactory general sampler exists on the market for the one-dimensional case of forcefully ducted horizontal material streams. As one example, the international power industry is currently converting coal-fired power plants to be able to use biomass as fuel, especially in CHP installations. In this endeavour transportation of various pulverized biomass-derived fuel types from macerating mills to the combustion chamber are carried out via pneumatic transportation. Reliable on-line information about particle size, moisture and sometimes also for a few other chemical/physical characteristics are often of critical importance for the combustion efficiency and therefore the economy of the power plant.

New industry standards currently being developed stipulate that sampling at this process position must be able to document a relative accuracy below 5%. This limiting demand has naturally served as the ultimate guideline for the development of the E-F-sampler.

While sampling in vertical flow streams, especially falling streams, is a relatively easy task when applying TOS correctly, horizontally ducted flows moved under a confining pressure present a much more difficult sampling challenge, indeed for many decades considered an (almost) impossible task. Firstly, all horizontally transported material streams must per force cause vertical segregation. Counteracting this constant, potentially severe segregation possibility must therefore be one of the prime design objectives for the EF-sampler. Furthermore pneumatic transportation systems cannot be arbitrarily intersected for extracting increments, due to the risk of pressure loss and unwanted discharge of material. It is also important that the sampling process itself does not constrict the transportation flux in order minimize the risk of pressure surges or clogging of pipes.

In the near-future design phase, the current 1/-scale EF-sampler as presented here will be scaled up to fit standardized coal pipeline diameters and installed in a converted coal power plant now utilising biomass. A comprehensive long-term experiment will be set up to evaluate the behaviour of the sampler in this full-scale industrial environment.

The following (Figures 3, 4 and 5) show schematic overviews of the EF-sampler and its two main functioning sections: extraction and cleaning/storage.

The extraction mechanism consists of a scythe-shaped sampling arm, which rotates 180 degrees through the ducted flow stream around a vertical axis, as shown in the top view of (Figure 4). The direction of the sampling arm movement can be adjusted according to the material flow, so that both prograde and retrograde movements are possible (not necessary for many fixed industrial installations, but interesting for equally challenging other, propriety application scenarios under parallel development). An electric power supply with sufficient over-capacity has been installed to facilitate a constant rotational velocity of the sampling arm, no matter what flux encountered. By using this efficient power source, acceleration and deceleration time of the engine and sampling arm respectively have also been minimized. Two sensors on each parking side control the correct resting position of the sampling arm. In the unlikely case that the sampling arm gets stuck in the material flow, the position sensors detect this error immediately and stop the sampling operation so that no further material is extracted and remedial actions can be initiated.

To clarify the half-circular movement of the sampling arm, the material flow direction has been marked with arrows in (Figure 4 and 5). The grey box-shaped area on top of the left (Figure 4) represents the power supply in the first prototype, which has since been modified in the final prototype and is now facing downwards (compare Figure 3). The dashed rectangle in the left top view of (Figure 4) depicts the parking position of the sampling arm, i.e. the position of the sampling arm while being inactive. Due to a complete 180-degree movement through the ducted flow for each increment extraction step, the sampling arm can rest also on the opposite side until moving back through the material flow to its initial position. When in either of these symmetrical parking positions the sampling arm is not interfering with the transportation flux. It has been a crucial requirement that the sampling arm should not constrict the material flow, neither while sampling nor while resting in the parking position. By this requirement the risk of clogging of material as well as for pressure surges has been minimized.

These general issues will to some degree always be dependent upon the specific material being transported and its aggregate characteristics. As an example, it is obvious that a generally fibrous material will behave differently from a generally grain-aggregate material, the latter being very much easier to sample than the former. It has been clear from the outset of the design that a completely general sampler could not, indeed should not, be realised. However, many related types of material should be amendable for sampling with only standard changes and modification regarding the intrinsic dimensions of the scythe arm, e.g.

cutter width, depth etc. Also the cross-cutting velocity can be changed without any effort via a simple computer command.

The sampling arm has been designed with the primary objective of reliably sampling the complete vertical segregating slice of the material stream, be this segregation minor, significant, or severe. A detailed drawing of the sampling arm design features is shown in (Figure 5, right). The sampling arm consists of a u-shaped "box" with two parallel sidewalls, termed the 'cutter' hereafter. Depending on the size distribution characteristics of the material to be sampled, the opening width of the cutter must accommodate the requirement of being at least three times the nominal top diameter of the ducted material in order to prevent clogging [1,2]. Furthermore the cutter must have a sufficient volume (depth) depending on sampling speed and material flow rate, to completely eliminate any risk for over-spilling of material during the sampling operation. The outer angle of the two parallel cutter blade tips should have an angle of at least 70 degrees to prevent material not belonging to the delineated increment from 'climbing' up along the edges into the cutter [2,15]. The cutter blades of the sampling arm are designed to be analogous to parallel cutter blades on conventional cross-stream samplers, which have been proven to delineate increments correctly, ibid. Deviations from these design requirements would lead to both Increment Delimitation Errors (IDE) and Increment Extraction Errors (IEE), therefore contributing to an inconstant sampling bias.

The downward facing outlet chute of the sampling arm (rectangle marked '26' in (Figure 5, left)) is used for isokinetic extraction of the material captured in the cutter arm into the storage section of the sampler. In order to prevent material not belonging to the delineated increment of ending up in the sample during the sampling operation, the extraction opening is covered with a shielding plate when in the parking position (Figure 5, right).

Beneath the outlet chute a three-way valve is installed (Figure 7). During the active sampling operation the valve is opened, so that the delineated material can fall into the upper storage cylinder, termed the 'compositing cylinder' (Figure 3 and 7). While the sampler is inactive, in the parking position, the valve is closed, preventing material falling through this extraction tube into the composite cylinder. The third valve position is activated before the cutter arm can be cleared to start its active sampling movement. In this position, pressurized air is blown upwards through the outlet chute into the sampling arm, facilitating removal of unwanted material in the cutter before taking the next increment. After the required increments has been extracted and collected in the composite cylinder, the three-path valve is again set to the closed position and the sampling cycle is finished.

Before the storage valve, which is situated below the composite cylinder (Figure 7), can be opened in order for the material to fall from the compositing cylinder into the final storage container, the pressure in the composite cylinder has to be equalised to ambient pressure. For this purpose an additional valve with a filter is installed on the top of the storage cylinder, allowing to reduce the remaining pressure in the compositing cylinder without loosing fines. The final storage cylinder has been designed in such way that it can be easily exchanged and sealed directly after removal.

A PLS steering unit controls the entire sampling process. The number of increments to be used for composite sampling, rotation/cutting speed, pause interval, as well as cleaning duration must be defined before the sampling operation starts. Each of these parameters can be separately set in a predefined range by a software user interface. After starting the sampling unit, the cleaning mechanism is always activated first in the predefined time period, followed by the opening of the three-path valve, which commences the actual sampling operation. Once the position sensor detects that the sampling arm has completed a 180-degree movement, the three-path valve below the outlet chute is closed and the predefined pause interval starts. This course of action (cleaning, sampling, and pausing) is repeated until the required number of increments has been extracted.

Estimating the required number of increments to be composited is a critical success factor for any process sampling operation, described many places in the relevant literature, see general process sampling references below. Most recently this issue was detailed in Esbensen et al., Minkkinen et al. and Esbensen et al. [10,11,12]. For significantly heterogeneous materials the authors arrive at appropriate number of increments between 42 and 100. In the prospective industrial setting the driver will always be optimal accuracy and precision, so the present testing phases resolved to use 75 increments, which is a reasonable projection from the full-scale industrial context, in which a representative sample will be required with a minimum resolution of 5 minutes (Vattenfall DK, DONG ENERGY pers. com.).

Experimental Test Design

All testing was carried out on a semi-industrial pneumatic transportation test system situated at the Department of Powder Science and Technology, Tel-Tek in Porsgrunn, Norway. A schematic overview of the full round pneumatic transportation system is depicted in (Figure 6), comprising a total distance of approximately 30 meters. The scale-version of the presented EF-sampler has been designed to fit the 3-inch piping system with pipe wall thickness of 3mm.

Before each transportation round, the material is filled from the receiving tank into the feeding tank. Below the feeding tank a rotational feeder is located (not depicted in (Figure 6)) allowing to adjust the feeding rate of the material into the piping system. A vibrating mechanism attached to the external wall of the feeding tank facilitates a constant material flux into the rotational feeder. By controlling the amount of compressed air ducted into the piping system, the dilution of the transported material, i.e. the flow regime, can be set to the required flow characteristics. The 'X' in (Figure 6) marks the installation position of the EF-sampler. For minimizing the effect of flow disturbances, mainly 'roping effects', the sampler position was chosen to be at least 3m away from any pipe bends (distance to previous bending was ca. 7m). The roping effect describes a particle segregation effect in pipe bends caused by the action of centripetal forces, whereby particles flowing to the outer wall of the bend form a relatively dense phase structure, termed rope [16].

Pneumatic conveying can be broadly categorized in dense phase vs. dilute/lean phase conveying. Despite the fact that the literature lacks a clear separation between these flow regimes, dilute phase can be best understood as a flow state where solid particles are fully suspended in the air or gas stream and behave as individual dynamic units, in contrast to dense phase where particles are severely influencing one-another in the aggregate stream flux, i.e. definitely not fully suspended [17]. Depending on the application, each flow regime carries its own advantages and disadvantages. The following experimental test campaign has been conducted with varying flow rates commensurate with the dilute phase regime only, since the EF-sampler was designed primarily for dilute phase biomass conveying (but with an eye towards more general applicability of course). One main advantage of dilute

phase conveying in terms of representative sampling is the fact that vertical flow segregation is lower than in a dense phase flow. The results section clarifies this aspect more fully.

Figure 7 shows the EF-sampler with its main technical components as installed at the marked position on the pneumatic conveying system (compare Figure 6). The left photograph shows the extraction and upper storage section, while the right illustration depicts a close-up of the lower storage section.

For the all-important verification of accuracy and representativeness, sampling tests with two very different materials were performed: wheat flour and pulverized alumina. Wheat flour has been selected to represent a very fine, cohesive powder, similar to cement powder and fly ash for example, two further powders often conveyed in pneumatic systems. The second test material, alumina, was selected since presenting a quite dense material type but with good flow characteristics. The present campaign aims at a first general feasibility testing for which these two materials were deemed sufficient; a follow-up campaign with several other, market-relevant, materials is in progress.

Particle size distribution, material density as well as the total amount of conveyed materials is presented in (Table 2). In order to focus on validation of accuracy and precision, spiking material in predetermined precisely calibrated concentration levels was added to each of these materials. LDPE (Low Density Polyethylene) plastic pellets were selected as a well-near optimal spiking material, having a density in-between the two test materials and, crucially, a particle size which is significantly larger than both test materials, i.e. a significantly different minor to trace constituent. The concentration levels of LDPE pellets in wheat flour were set to 0%, 2% and 5% [w/w] respectively. For the subsequent alumina test campaign the concentration level range was expanded to 0%, 2%, 5%, 8% and 11% [w/w]. The physical characteristics of the plastic pellets are also stated in (Table 2). It was our deliberate intention to present the sampler with materials of a reasonably high degree of difficulty, for which reason wheat and alumina powders with added LDPE pellets actually constitute materials that are more difficult to sample that the nominated biomass target, when in routine transportation in the designated industrial setting. For feasibility and validation purpose however, this stringent test scenario will serve very well however.

Test material	Density	Particle size distribution			Amount of material
		D10	D50	D90	
Wheat flour	0.46g/cm^3	13.3µm	66.5µm	161.3µm	240kg
Alumina	1.25g/cm^3	35.7µm	85.7µm	134.5µm	150kg
Plastic pellets	0.58g/cm^3	~3mm			Depending on concentration level

Table 2: Physical characteristics of test materials.

Test material	Naming of flow rate	Mass flow (kg/s)	Airflow (Nm3/h)	Mass-air-ratio
Wheat flour	FR 1	0.30	750	$\frac{1.2}{1}$
	FR 2	0.20	950	$\sim\frac{0.6}{1}$
Alumina	FR 1	0.50	750	$\frac{2}{1}$
	FR 2	0.35	950	$\sim\frac{1}{1}$

Table 3: Tested mass/air ratios of wheat flour and alumina.

Test material	Naming of sampling speed	Sampling speed (s)	Pausing time (s)	Cleaning time (s)
Wheat flour	SP 1	5	1	2
	SP 2	3	3	2
Alumina	SP 1	2	1	1
	SP 2	1	2	1

Table 4: Test parameters of EF-sampler.

Test material	Spiking concentration levels (%)	Flow rates	Sampling speed	Repetitions
Wheat flour	0, 2, 5	FR1, FR2	SP1, SP2	3
Alumina	0, 2, 5	FR1, FR2	SP1, SP2	2
	8, 11	FR1, FR2	SP1	2

Table 5: Overview of complete test campaign parameters.

RSV (%)	FR1, SP1	FR1, SP2	FR2, SP1	FR2, SP2
Wheat flour - 2% spiking	2,91%	2,63%	4,24%	4,04%
Alumina – 5% spiking	2,18%	2,98%	1,91%	3,43%
Alumina – 11% spiking	4,22%	-*	2,53%	-*

* These scenarios have been only tested with sampling speed 1.

Table 6: RSVs of the present test scenarios.

In order to validate whether variations of the mass-air ratio in the dilute phase regime affects the sampling results, the airflow was varied between 750 Nm^3 (Flow rate 1) and 950 Nm^3 (Flow rate 2) with a constant feeding rate for both test materials; a constant feeding rate ensures an equal volume flow of both test material into the piping system. Due to the higher density of alumina an identical constant feeding rate results in a higher mass-air-ratio for alumina when varying only airflow in the different test scenarios. (Table 3) depicts the realised mass- and airflow as well as the resulting mass-air ratios in the test scenarios for both materials.

The test campaign also comprised variations in sampling speed (rotational speed of cutter arm) as well as adjusting cleaning- and pausing times in order to allow that 75 increments could be extracted in the required time for each transportation round. Due to the different test material load realised, two sampling speeds were set for wheat flour and correspondingly for alumina. (Table 4) lists the varying sampling speeds and cleaning-/pausing times for each test material.

For all scenarios a sampling arm width of 14 mm was used with a depth of 15 mm ensuring a correct delineation of the increment and preventing any spill-over effects. For test materials with a larger nominal top diameter, the sampling arm needs to be adjusted accordingly. As a rule of thumb TOS states that the width of the cutter or sampling arm should be at least three times the nominal top diameter of the material of interest. The E-F sampler is designed so that replacement of the sampling arm can be carried out without undue efforts, i.e. without having to detach the sampler in its pipefitting a.o.

A complete overview of all test parameters including replication rounds is stated in (Table 5), resulting in a total of 36 test rounds for wheat flour and 32 test rounds for alumina. It is important to mention that in each transportation round the entire test material (lot) was transported from the feeding tank into the receiving tank, obeying TOS' principle of sampling correctness for the entire lot. The required amount of plastic pellets presenting different nominal spiking concentrations levels was inserted directly into the feeding tank, while discharging the test material from the receiving tank into the feeding tank. Since a vestige of a layering effect of the plastic pellets could not be completely avoided during insertion, the spiked material was

transported passively for two full circulation rounds before starting the sampling test operations proper in order to achieve an effective 'in-line' mixing effect. By this type of successively sampling of the lot, the remaining segregation effects in the feeding tank could be minimized, and perhaps even fully counteracted. Any such lingering effects will of course show up in the test results, i.e. as inflated accuracy and precision measures, but as long as these stay within the pre-specified brackets this will be an acceptable price to pay for a sampler that can accommodate the highly taxing pneumatic horizontal flow scenario.

Results

In the following sections selected results of the test scenarios are presented and discussed. Besides estimation of the effective accuracy and precision range of the EF-sampler under the applied test conditions, the experimental results also show to which degree the predetermined test parameters influence upon these evaluation criteria. The range of each test parameter had to be pre-set before the actual test campaign. This implies that the globally optimal set up of test parameters may actually be found (with more experimentation) lie outside this initial test parameter range. Because of the feasibility test objective, it was considered acceptable to try to shoot for the critically important accuracy testing with a manageable set if inferred best test conditions first. Below are presented selected results, not all corresponding to what was eventually found, after all experiments were in, to be the optimal conditions.

(Figure 8) depicts the sampling test results for wheat flour, spiked with a plastic pellet concentration of 2%. The left figure shows sampling results using 0% pellet concentration as the origin on the y-axis origin, while the right figure shows a close-up of the variations in the occurring concentration level range. The y-axis displays the plastic pellet concentration of composite samples consisting of 75 increments. The x-axis illustrates the mean value concentration of the three replication rounds for each test scenario. On the left side of the y-axis sampling results for flow rate 1 (FR1) and flow rate 2 (FR2) using sampling speed 1 (SP1) are compared, while on the right side of the y-axis analysed concentration values of the samples gained from FR1 and FR2 with sampling speed 2 (SP2) are shown. Furthermore the nominal reference concentration level is depicted as a dashed line.

For these initial test scenarios all composite samples with respect to the analysed spiking concentration level have a relative inaccuracy smaller than 16% (highest rel. inaccuracy occurring in test scenario W2_FR1_SP2). For both sampling speeds (SP1 and SP2), a higher airflow rate (FR2) leads to an improved accuracy of the plastic pellet concentration in the wheat flour, while the longer sampling time 1 (SP1=5s) further improves the accuracy compared to the shorter cutting interval SP2 (sampling speed=3s).

Thus the highest accuracy for this scenario, rel. accuracy <4,5%, was achieved by using SP1 with FR2, i.e. the longer sampling time in the more dilute material stream. These results also point out that nearly all analysed concentration levels of the composite samples lead to a slightly higher concentration compared to the reference level of 2%. An explanation for this bias is given after presenting selected results for the alumina test scenarios. The precision for all test variations is good throughout.

(Figure 9) presents the results of composite samples extracted during the pneumatic transportation of alumina with a plastic pellet concentration of 5%. Each test scenario has again been repeated twice, varying sampling speed (SP1, SP2) and airflow rate (FR1, FR2).

The worst (highest) relative inaccuracy for the alumina tests is now below 11%, occurring in the composite sample gained with the faster sampling speed (SP2 for alumina=1s) and the lower airflow rate 1 (FR1). The close-up of (Figure 9) confirms that composite samples extracted during a higher airflow rate are much more accurate (rel. inaccuracy <4%), being further improved by the slower sampling speed SP1 (SP1 for alumina=2s). These observations are consistent with the results outlined by the initial wheat flour scenarios above. The positive effect of longer sampling times (SP1) can easily be explained by TOS, substantiating that an increase of the increment volume improves the accuracy of the composite sample.

In contrast to the plastic pellet concentration analysed in the wheat flour experiments, which are slightly overrepresented (also valid for 2% spiking concentration), the concentration levels in the alumina are slightly unrepresented (also valid for all other tested concentration levels). This effect can be also observed in (Figure 10), presenting the concentration levels for alumina spiked with 11% plastic pellets. For this scenario, which was repeated twice, only the slower sampling speed 1 (SP1) was used for increment extraction. The composite samples extracted during testing with the higher airflow rate 2 (FR2) lead again to a better accuracy, <4% rel.

Thus for both test materials, under the best sampling conditions obtainable (still sub-optimal with respect to the intended industrial conditions), a relative accuracy level below the pre-set 5% was indeed achieved under the test conditions available at the test site.

The corresponding lowest total sampling errors, including all sampling errors and total analytical errors, can be expressed as a convenient RSV (Relative Sampling Variance) measure[2], also termed the relative coefficient of variation (CV_{rel}). The CV_{rel}, meaning the standard deviation (STD) in relation to the average (X_{avr}), can be effectively expressed as a percentage:

$$CV_{rel} = \frac{STD}{X_{avr}} * 100$$

A CV_{rel} corresponding to 20-35% has been suggested for 'significantly heterogeneous materials', in particular for uncharacterized stationary systems [18], while for less heterogeneous systems, as also being the case for the presented test campaign, the CV_{rel} level should be set to a level specified as 15-20% [15].

Compared to these general guidelines, it is also the opinion that for process sampling these thresholds must be lower still due to the much more optimal sampling conditions that can be realised in this regimen. Due to the limited replication rounds of the present test scenarios, the results allow serve as first estimates, which are reported in (Table 6). These statistical results should optimally be based on a repetition of at least 10 repetition rounds, which was out of the possible range of test conditions available in this first trial in which the accuracy test had absolute priority. A full second test campaign on several additional material types and with further replications is under way.

All test scenarios have a relative sampling variance <4,5%, lying far below the suggested thresholds. Seen together with the <4.5% inaccuracy results, the RSV estimates show that the incorrect sampling

[2]Because only two, or three, replications could be achieved in the present test campaign, estimating a standard deviation on this basis is naturally a somewhat contrived endeavor. But if a first estimate of the relevant RSV is wanted, these have been calculated and reported in the text. They should be viewed in this particularly bracketed context of course.

errors in the EF-sampling system, the major source for bias and high sampling variance, have been very nearly eliminated, confirming the correctness of the EF-sampler's design principles as well as its performance.

Discussion

An explanation for the small bias detected for both test materials, over-representing the plastic pellet concentration in the wheat flour and under representing the concentration level in the alumina, can logically be attributed to the density differences between wheat flour, plastic pellets and alumina respectively, as was documented in (Table 2). Even though the sampling arm has been designed with the stated objective of being able to cope with the vertical segregation occurring in horizontal transportation sections, the test facility parameters do not allow to achieve a completely unbiased suspension of the compound mixed material; in the test facility used, the realisable air capacity and pipe dimension limit the dilution range possible. This means that it can be expected that the material with a higher density is (slightly) more likely to be transported at the bottom part of the piping system. Assuming an equal influx of material into the sampling arm, the more dense material most likely disturbs the less dense material flow inside the sampling arm, allowing a slightly higher proportion of dense material to fall directly into the outlet chute, located at the bottom of the sampling arm. This is of course a classical segregation effect in the framework of TOS.

Normally only one material type is transported in industrial pneumatic transportation rigs. The ultimate purpose of acquiring representative samples from CHP plant pneumatic transportation ducts is to assess the % of "fines" (the smallest biomass particle sizes). This scenario is thus not affected by the kind of density differences employed in the current test campaign, which were set up here for maximal test validity, i.e. the deliberate choice to test based on added minor contaminant concentrations of significantly different particle size as well as density with respect to the matrix material.

The obtained results show quite satisfactorily that the EF-sampler, even under these "more-than-necessary difficult" sampling conditions, extracts fit-for-purpose samples with performance results that can be considered as acceptably close to the pre-set acceptable accuracy threshold of 5%. The present results also reveal that higher airflow rate capacities would minimize the detected bias level. In the target case of converted coal power plants mass-air ratios up to 1 (mass) to 2 (air) are achieved, resulting in a very dilute flow regime favouring even better sampling accuracy. In a forthcoming comprehensive test campaign the EF-sampler will be tested under this very dilute flow regime in a fully up-scaled version for direct implementation in converted coal power plants, pneumatically transporting pulverized biomass from the mills to the combustion chamber. These results will serve to validate the performance of the EF-sampler under fully realistic industrial conditions, including larger process variations and varying compositions of the transported material.

For this up-scaled test campaign of the EF-sampler and also for any other sampler verification, the Theory of Sampling points out that the ultimate test regarding intrinsic parameters (e.g. chemical composition, physical grain-sizes a.o.) is to which degree a sampler is able to reproduce the lot grain size distribution quantitatively within the specified acceptance levels. Thus if the results with respect to the particle size distribution show an acceptable small bias (e.g. 5% rel. for all size bins), it follows that the EF-sampler is also similarly acceptably 'unbiased' with respect to most other chemical attributes. Powder characterises like cohesiveness or moisture level variations are parameters, which need further investigation in respect to their influence on the sampling results however, as these are not intrinsic characteristics. Furthermore, the optimal sampling speed for the set flow regime of pulverized biomass in converted power plants will be determined applying the results of the planned further test campaigns.

Conclusions and Prospects

The EF-sampler is the first sampler for horizontal pneumatic transportation systems, with the objective to ensure acceptable TOS-representativeness of samples extracted from ducted pressurised material streams. The design principles prevent that neither the sampling operation itself nor the resting of the sampler in its inactive parking position cause any major disturbances of the material flux, which could otherwise lead to pressure surges or clogging effects. The automated extraction mechanism, including the possibility to vary sampling-, cleaning- and pausing intervals makes the EF-sampler flexible and adjustable to many different material types and flow characteristics. However the sampling arm width must be commensurate with the grain size distribution of the material of interest. For the test scenarios presented a sampling arm width of 14mm was sufficient.

The first experimental test campaign allowed full validation of the technical functionality, the accuracy (bias) and the precision of the EF-sampler under varying conditions for the most influential parameters. Sampler velocity as well as the material dilution ranges influenced significantly on the accuracy of composite samples. Particularly an increase of airflow capacity leading to a higher dilution of the transported spiked material improved the sampling accuracy. The higher dilution of the spiked test material in combination with a slower sampler velocity resulted in a relative inaccuracy for all tested scenarios below 5% with a generally very good reproducibility (precision), i.e. fully compliant with the pre-trial critical success criterion established. Furthermore, the relative sampling variance for all test scenarios is quantitatively also below 5% (<4,5%), signifying that perhaps all critical sampling errors have been successfully eliminated or minimized.

The prototype EF-sampler for horizontal ducted suspended material streams is not a universal sampler, but is designed to incorporate material-dependant adjustments based on the specific material heterogeneity and flow regime characteristics. The present feasibility validation of the EF-sampler installed in a pilot-scale transportation system showed however that variation of spiking concentration, dilute flow range and sampling speed are fully controllable parameters, which in all test scenarios lead to fit-for-purpose representative sampling. The EF-sampler has thus passed the first crucial test qualifications with respect to its intended primary biomass implementation scenario, and with a promising much wider potential application field as well. It will be highly relevant to test it on a wide range of other market-relevant material types. Such testing is in progress and will be reported elsewhere.

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References

1. Gy P (1998) Sampling for Analytical Purposes. John Wiley and Sons Ltd, Chichester, UK.
2. Pitard FF (1993) Pierre Gy's Sampling Theory and Sampling Practice (2 edition), CRC Press Ltd, Boca Raton, USA.
3. Esbensen KH, Minkkinen P (2004) Special issue: 50 years of Pierre Gy's theory of sampling: proceedings: first world conference on sampling and blending (WCSB1). Tutorials on sampling: theory and practice. Chemometrics and Intelligent Laboratory Systems 74: 236, 1242.
4. Petersen L, Dahl CK, Esbensen KH (2004) Representative mass reduction in sampling-a critical survey of techniques and hardware. Chemometrics and Intelligent Laboratory Systems 74: 95-114.
5. Petersen L, Minkkinen P, Esbensen KH (2005) Representative sampling for reliable data analysis. Theory of sampling. Chemometrics and Intelligent Laboratory Systems 77: 261-277.
6. Petersen L, Esbensen KH (2005) Representative process sampling for reliable data analysis-a tutorial. Journal of Chemometrics 19: 625-647.
7. Esbensen KH, Julius LP (2009) Representative sampling, data quality, validation-a necessary trinity in chemometrics. In Brown S, Tauler R, Walczak R (Eds.) Comprehensive Chemometrics, Wiley Major Reference Works 4: 1-20.
8. Wagner C, Esbensen KH (2012) A critical review of sampling standards for solid biofuels-Missing contributions from the Theory of Sampling (TOS). Renewable and Sustainable Energy Reviews 16: 504-517.
9. Minkkinen PO, Esbensen KH (2010) Grab vs. composite sampling of particulate materials with significant spatial heterogeneity-a simulation study of "correct sampling errors". Anal Chim Acta 653: 59-70.
10. Esbensen KH, Paoletti C, Minkkinen P (2012) Representative sampling of large kernel lots-I. Theory of Sampling and variographic analysis. Trends in Analytical Chemistry (TrAC) 32: 154-164.
11. Minkkinen P, Esbensen KH, Paoletti C (2012) Representative sampling of large kernel lots-II. Application to soybean sampling for GMO control. Trends in Analytical Chemistry (TrAC) 32: 165-177.
12. Esbensen KH, Paoletti C, Minkkinen P (2012) Representative sampling of large kernel lots-III. General Considerations on sampling heterogeneous foods. TrAC Trends in Analytical Chemistry 32: 178-184.
13. Smith PL (2001) A Primer for Sampling Solids, Liquids and Gases-Based on the Seven Sampling Errors of Pierre Gy. ASA SIAM, USA.
14. Wagner C, Faust H, Esbensen KH (2011) Proceedings of 5th World Conference on Sampling and Blending (WCSB 5). Experimental testing of 1/3 scale model sampler for horizontally ducted particulate material streams 445-451.
15. Pitard FF (2009) 'Pierre Gy's Theory of Sampling and C.O. Ingamells' Poisson Process Approach. Pathays to representative sampling and appropriate industrial standards. Dr. Tech Thesis Aalborg University, Campus Esbjerg, Denmark.
16. Akilli H, Levy EK, Sahin B (2001) Gas-solid flow behaviour in a horizontal pipe after a 90˚ vertical to horizontal elbow. Powder Technology 116: 43-52.
17. Fokeer S, Kingman S, Lowndes I, Reynolds A (2004) Characterisation of the cross sectional particle concentration distribution in horizontal dilute flow conveying-a review. Chemical Engineering and Processing 43: 677-691.
18. Esbensen KH, Geladi P (2010) Principles of proper validation: use and abuse of re-sampling for validation. Journal of Chemometrics 24: 168-187.