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# Particle size, size distribution and morphological evaluation of glass fiber reinforced plastic (GRP) industrial by-product

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ABSTRACT

The waste management of glass fiber reinforced polymer (GRP) materials, in particular those made with thermosetting resins, is a critical issue for the composites industry because these materials cannot be reprocessed. Therefore, most thermosetting GRP waste is presently sent to landfill, in spite of the significant environmental impact caused by their disposal in this way. The limited GRP waste recycling worldwide is mostly due to its intrinsic thermosetting properties, lack of characterization data and unavailability of viable recycling and recovery routes. One of the possibility for re-using GRP industrial by-product is in form of powder as a partial aggregate replacement or filler addition in cement based composites for applications in sustainable construction materials and technologies. However, the feasibility of this kind of reutilization strongly depends on the morphology and particle size distribution of a powder made up of polymer granules and glass fibers. In the present study, the use of image analysis method, based on scanning electron microscopy (SEM) and ImageJ processing program, is proposed in order to evaluate the morphology of the particles and measure the particle size and size distribution of fine GRP waste powder. The obtained results show a great potential of such a method in order to be considered as a standardized method of measurement and analysis in order to characterize the grain size and size distribution of GRP particles before exploiting any compatibility issue for its recycling management.



## 1. Introduction

Sustainable building development includes a wise management of resources, achieved by the use of industrial by-products and post-consumers discarded materials, and a lower environmental impact achieved through reduced natural aggregate mining from quarries (Naik, 2007; Malhotra, 2003). Concrete could be a feasible solution to environmental problems since it is also possible to re-use solid by-products from other industries for concrete production. This would reduce the need to landfill these materials and to extract natural aggregate from quarries while still maintaining an acceptable concrete quality (Hendriks and Janssen, 2003). In particular, for self-compacting concrete production, a high-volume of very fine material is also necessary in order to make the concrete more fluid and cohesive. Self-compactability of concrete is generally achieved by adding a certain amount of very fine particles as a filler to the mixture.

Glass reinforced plastic (GRP) is a composite material made of glass fibers dispersed in a resin, usually polyester, widely used in several fields from building, furniture and boats. GRP materials are being increasingly used also in the construction and transportation industries. The composites industry is now producing a wide range of GRP products that include strengthening strips and sheets, reinforcing bars, structural profiles, sandwich panels, molded planks and piping. During GRP products manufacturing, polyester resin is reinforced with glass fibers to obtain a lightweight building material showing excellent mechanical performance, particularly stiffness and tensile strength. The GRP manufacturing process produces considerable quantities of unfinished products as waste materials. Additionally, GRP waste materials have been often disposed off by the construction, automobiles, locomotives and aerospace industries after their utility/service life is over (Asokan et al., 2009). GRP waste is basically a combination of glass fibers and polymer and disposal of such solid waste to landfill becomes a major environmental hazard. GRP produced in Europe in 2010 amounts to 1,015,000 tons and in Italy to 154,000 tons. GRP disposed of in 2008 in Italy amounts to:

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CER 070213 (plastic waste): 159,289 tons;
CER 101103 (waste from glass fiber based materials) 26,763 tons;
CER 120105 (plastic materials leftover curls) 135,241 tons (UCINA, 2012).

In Italy the above by-products are landfilled due to the difficulty of separating the glassy part from the polymeric matrix, their intrinsic thermoset composite nature, the lack of information relating to their characteristics and the insufficient knowledge on potential recycling options. A number of studies have been carried out dealing with the use of recycled glass (Alia and Al-Tersawy, 2012; Kou and Poon, 2009) and polymeric addition (Jo et al., 2008; Siddique et al., 2008) in civil engineering. The feasibility of re-using GRP industrial by-product in manufacturing cementitious elements could be similarly considered (Tittarelli and Moriconi, 2010). In particular, three types of GRP by-products are presently produced:

- GRP bars: 1 m long and 1–2 cm thick, coming from faulty products;
- small pieces: 1–2 cm in size;
- a fine powder: about 0.1 mm in size.

The reutilization of the fine GRP powder as a partial cement replacement (Tittarelli et al., 2004; Tittarelli and Moriconi, 2005) or partial fine aggregate replacement (Tittarelli and Moriconi, 2010) or filler addition for self-compacting concrete (Tittarelli and Moriconi, 2008), as well as its influence on the durability of the cementitious products in which has been applied (Tittarelli and Moriconi, 2004), has been already widely exploited. However, contradictory results on the technological advantage of this reutilization, consisting of lower risk of cracking induced by restrained drying shrinkage and significantly lower capillary water absorption (Tittarelli and Moriconi, 2007), are reported in the literature. Indeed, the durability improvement caused by the GRP powder reutilization strongly depends on the morphology and particle size distribution of a powder made up of polymer granules and glass fibers, so that an incorrect or misleading characterization of the GRP waste powder, mainly attributable to improper evaluation of the granules and fibers fractions in it, can produce contradictory results.

In the present study scanning electron microscopy (SEM) and ImageJ processing program have been used in order to evaluate the morphology of the particles and measure the particle size and size distribution of the fine GRP powder by-product in order to outline compatibility issues with cement from a dimensional point of view. The use of an image analysis method is proposed given that the use of indirect advanced methods, such as for example those involving scattered or diffracted light or laser, assumes the particle to be spherical, which is not the predominant case with GRP materials. The results obtained by using image analysis have been compared with the results obtained by standard size distribution technique such as for example laser diffraction particle size analysis.

It seems from the obtained results that image analysis shows a great potential to be considered as a standardized method of measurement in order to characterize the grain size and size distribution of GRP particles before exploiting any compatibility issue for its recycling management.

## 2. Materials and methods

A schematic flowchart of the various stages involved in size analysis is illustrated in Fig. 1.

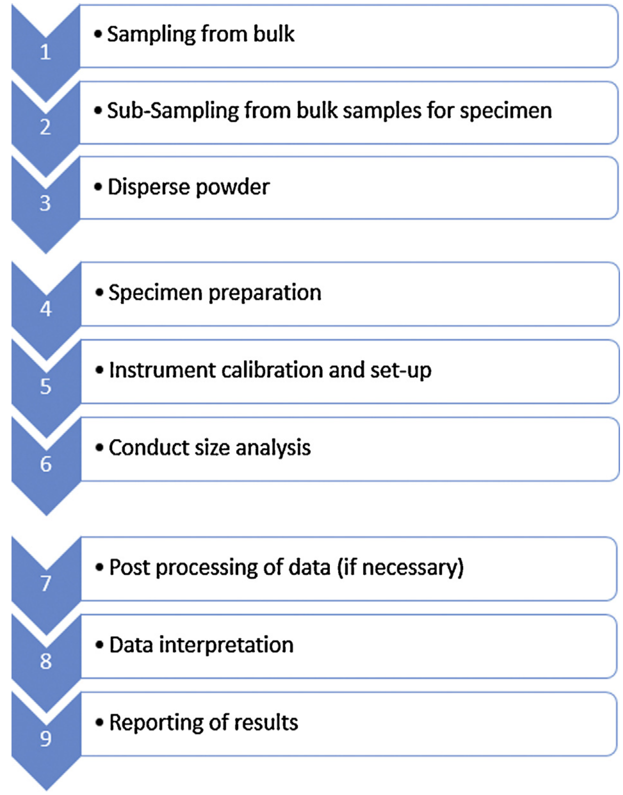


**Fig. 1.** Flowchart of general procedures followed for particle size and size distribution analysis.

### 2.1. Sample preparation for SEM analysis

A GRP powder coming directly from a shipyard as an industrial by-product was used. The samples were prepared for SEM analysis according to a specific previously described procedure (Mazzoli and Favoni, 2012). Samples of 20 mg were suspended in 20 ml of distilled water. The suspension was actively stirred, under sonication, in order to avoid dust aggregates. A fraction of 0.5 ml of the above suspension underwent gravity filtration. The filter was a polycarbonate membrane (Isopore Membrane Filters by Millipore) with a porosity of 0.4 μm. After the filtration the membrane underwent an air drying process. The central body of the membrane was then cut using a scalpel, fixed to the stab and gold coated under vacuum in order to ensure image quality during the SEM analysis. Images have been acquired using a SEM microscope Zeiss Supra40. The SEM process conditions were the following: depth of coating 30 nm and pressure $7 \times 10^{-2}$ mbar. Gravity filtration has been chosen because it strongly reduces, with respect to vacuum filtration, the possibility of preferential alignment of the particles to the pores of the membrane. The samples have been collected on three different membranes and the results mediated.

### 2.2. SEM analysis

As discussed in ISO 13322-1 the measurement of particle size distributions by microscopy methods is apparently simple, but due to the limited amount of sample examined, considerable care must be taken in order to ensure that the analysis is representative of the entire sample (Standard ISO, 2004). ISO 13322-1 recommends splitting the original sample and making measurements on three or more parts. For the above reason each stab has been ideally divided in four sectors and the relative images acquired. For each sample four images have been acquired, one for each sector of the

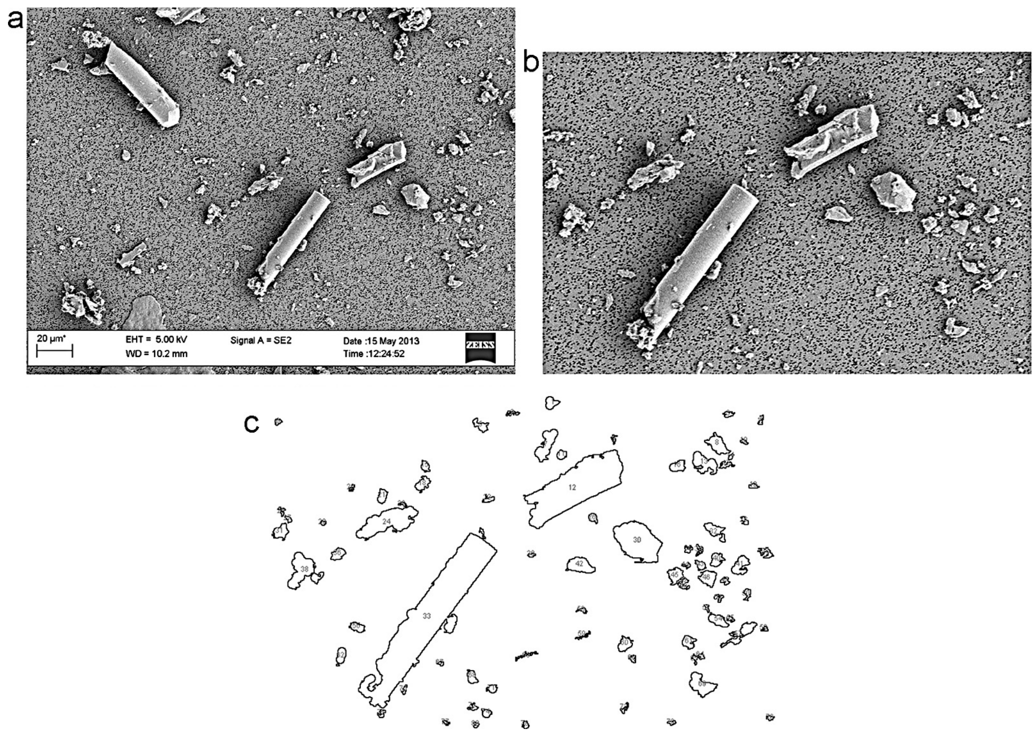


**Fig. 2.** (a) Original SEM micrograph; (b) selected portion of the micrograph for the analysis of the particles; (c) results obtained using ImageJ.

stab, assuring a minimum quantitative of particles for each sample according to the standard ISO 13322-1. Sample with GSD (geometric standard deviation) of 1.2 needs, in fact, 2939 particles to achieve mass median diameter within 5% error with 95% probability (Standard ISO, 2004).

### 2.3. Image processing and analysis

The obtained images were processed using the software ImageJ. ImageJ developed at the National Institutes of Health (NIH), USA, is a Java-based public domain image processing and analysis program, which is freely available, open source, multithreaded, and platform independent that can be utilized to develop user-coded plugins to suit the specific requirements of any conceived application (Rasband, 1997). The first step in using ImageJ is the image calibration required to correlate the image dimensions in pixel to physical dimensions. Image processing algorithms require a binary image that can be produced by converting the 8-bit image, that can display 28 gray levels, by proper thresholding. Proceeding with the correct threshold limits creates the required binary image. In fact, if the object is properly covered by thresholding, then accurate size measurement is automatically guaranteed as the image processing method is direct in principle. The procedure of measurement is obtained by running the ImageJ's "Analyze particles" routine. The options "Exclude on Edges" and "Include Holes" were selected in order to ensure including all whole particles and ignoring holes, if any after thresholding, in the particles of the image during analysis. In the below figures are reported, as an example, the original SEM micrograph (Fig. 2(a)), the selected portion of the micrograph chosen for the analysis of the particles (Fig. 2(b)) and the results obtained using ImageJ (Fig. 2(c)).

Given that GRP particles show an irregular shape, assuming them to be of regular geometrical shapes will result in oversimplified approximation. In fact, a spherical particle can be described using a single number – the diameter – because every dimension is identical. As can be seen in Fig. 3, non-spherical particles can be described using multiple length and width measures (horizontal and vertical projections are shown here). These descriptions provide greater accuracy, but also greater complexity. Thus, many techniques (i.e. scattered light, acoustic attenuation, settling rate) make the useful and convenient assumption that every particle is a sphere. The reported value is typically an equivalent spherical

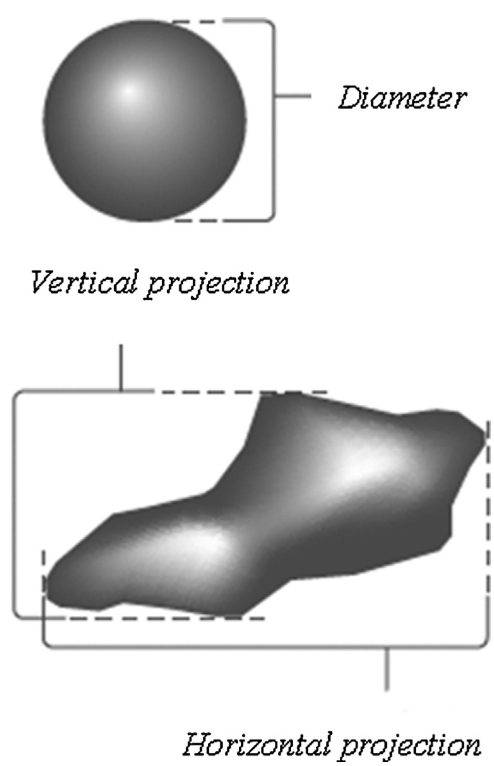


**Fig. 3.** Shape factor.

diameter. Microscopy or automated image analysis are the only techniques that can describe particle size using multiple values for particles with larger aspect ratios as described also by Gomez Yepes and Cremades (2011). An image analysis system such as ImageJ is able to describe the non-spherical particle seen in Fig. 2 by the longest and shortest diameters, bounding rectangle, fit ellipse, perimeter, projected area, or again by equivalent spherical diameter.

The measurement of particle sizes varies in complexity depending on the shape of the particle, and the number of particles characterized must be sufficient to ensure an acceptable level of uncertainty in the measured parameters. Additional information on particle size measurement, sample size and data analysis is available in ISO 9276 (Standard ISO, 1998). As for regards spherical particles, size is defined by the diameter. For irregular particles, such as the fibers and also a portion of the particulate, a variety of definitions of particle size exist. For the above reason in this paper the particles and the fibers have been separately analyzed. As for regards the particulate portion of the GRP powder the built-in standard measurements of ImageJ's "Analyze Particles...", such as diameter of a circle of equal projection area, Feret's diameter and minimal Feret's diameter were chosen also on the basis of previous papers found in the literature (Walton, 1948; Al-Thyabat and Miles, 2006; Pabst et al., 2006; Igathinathane et al., 2009). The diameter of a circle of equal projection area ($d_{EC}$) is the diameter of a circle that has the same area as the projection area of the particle as can be seen in Fig. 4(a). The Feret's diameter ($d_F$) is the longest distance between any two points along the selection boundary, also known as maximum caliper as can be seen in Fig. 4(b). The minimal Feret's diameter (Min $d_F$) is the minimal Feret's diameter calculated after considerations of all possible orientations (0°, ..., 180°).

As for regards the fibers contained in the GRP powder, they have been characterized using the Feret's diameter ($d_F$), the bounding rectangle and the fit ellipse measurements tools offered by ImageJ. The bounding rectangle is the smallest rectangle enclosing the selection. Fit ellipse fits an ellipse to the selection as can be seen in Fig. 5.

To estimate the accuracy of the machine vision method, a $TiO_2$ powder (Sigma–Aldrich, MO, USA), predominantly rutile showing a particle size less than 5 μm, was selected and analyzed using ImageJ as previously described in another paper of one of the authors (Mazzoli and Favoni, 2012). In order to evaluate the particulate and the fiber size distributions, 3487 particles were analyzed from electron micrographs and the value of the $d_{EC}$, $d_F$ and major and minor axes of the fit ellipse are reported. The results of a method that use diffracted laser, that assumes that the particle is spherical, applied to the GRP powder are also reported in order to demonstrate its unsuitableness in such a case.

## 3. Results

The samples of GRP powder have been collected on three different membranes and the results mediated. The particle size distribution can be calculated based on several models: most often as a number or volume/mass distribution. As for regards the particle size distribution of particulate the results, based on a number distribution, are reported in Figs. 6 and 7. The results are shown representing the percentage of particles in each size group on the basis of the $d_{CE}$ and $d_F$ values.

Building a number distribution has generated the result shown in the above reported figure. If this same result is converted to a volume distribution, the result would appear as shown in the figure below. In fact, the majority of the total particle mass or volume comes from the bigger particles. Nothing changes between the two graphs except for the basis of the distribution calculation. In Fig. 8

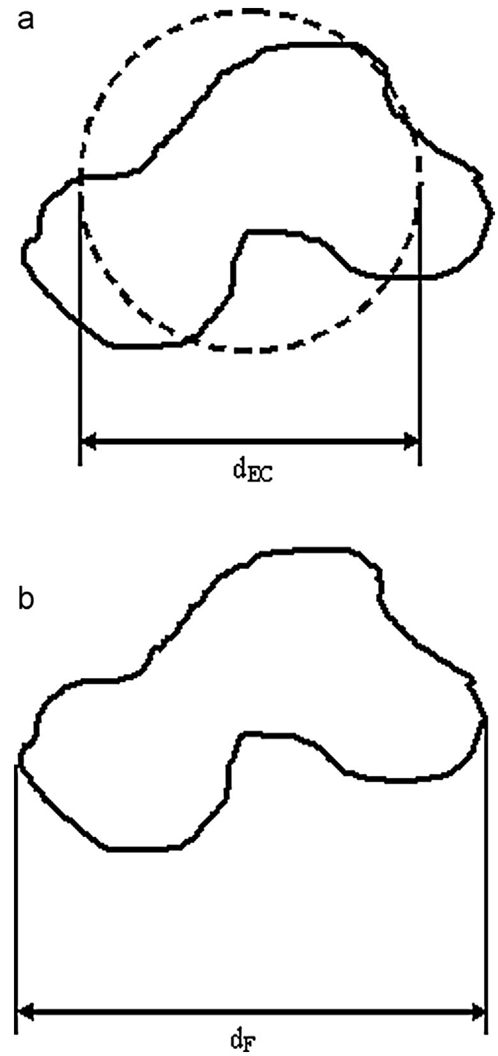


**Fig. 4.** Diameter definition. (a) Diameter of a circle of equal projection area ($d_{EC}$). (b) Feret's diameter ($d_F$).

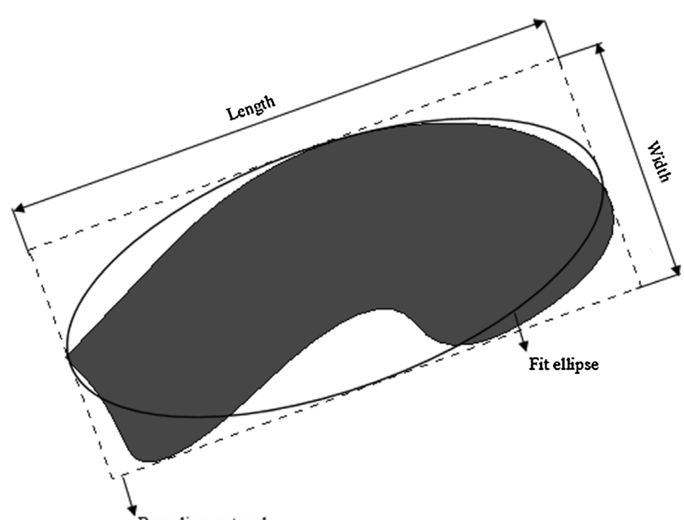


**Fig. 5.** Bounding rectangle and fit ellipse to an irregular shape.

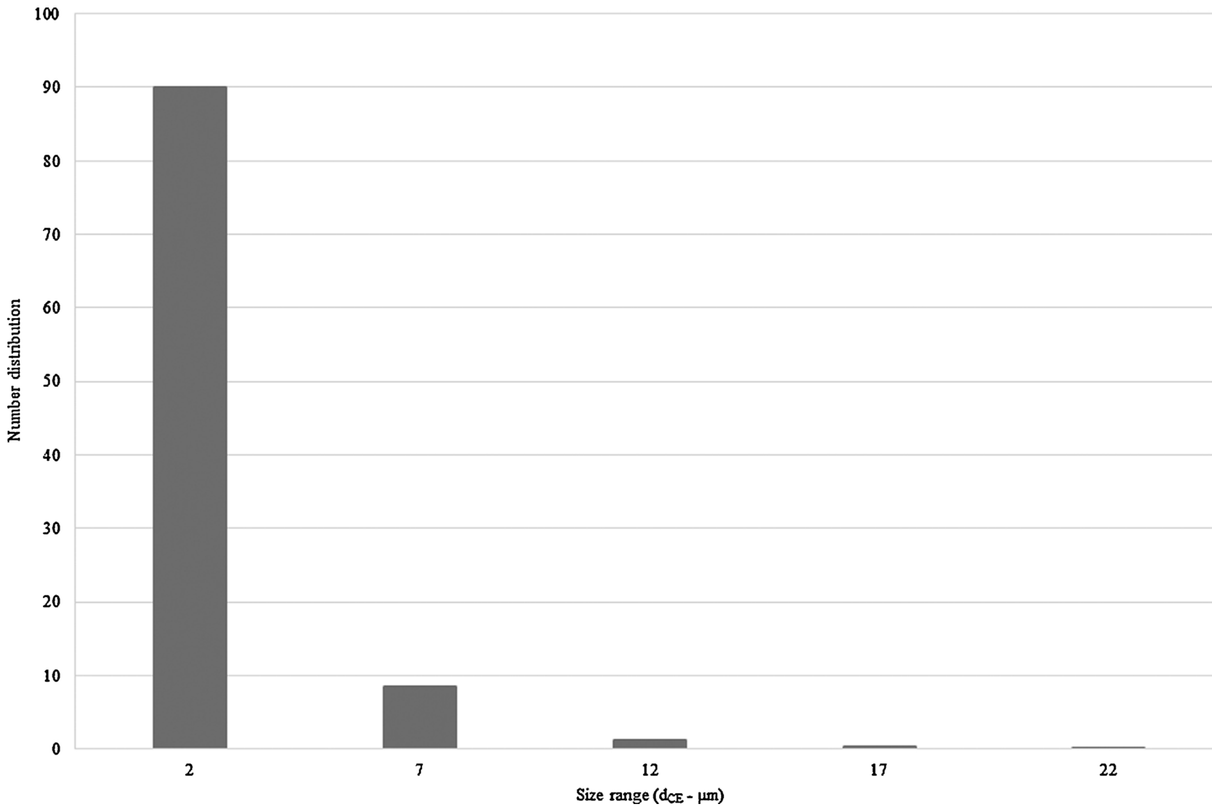


**Fig. 6.** Analysis of the particulate. Number distribution vs. $d_{CE}$.

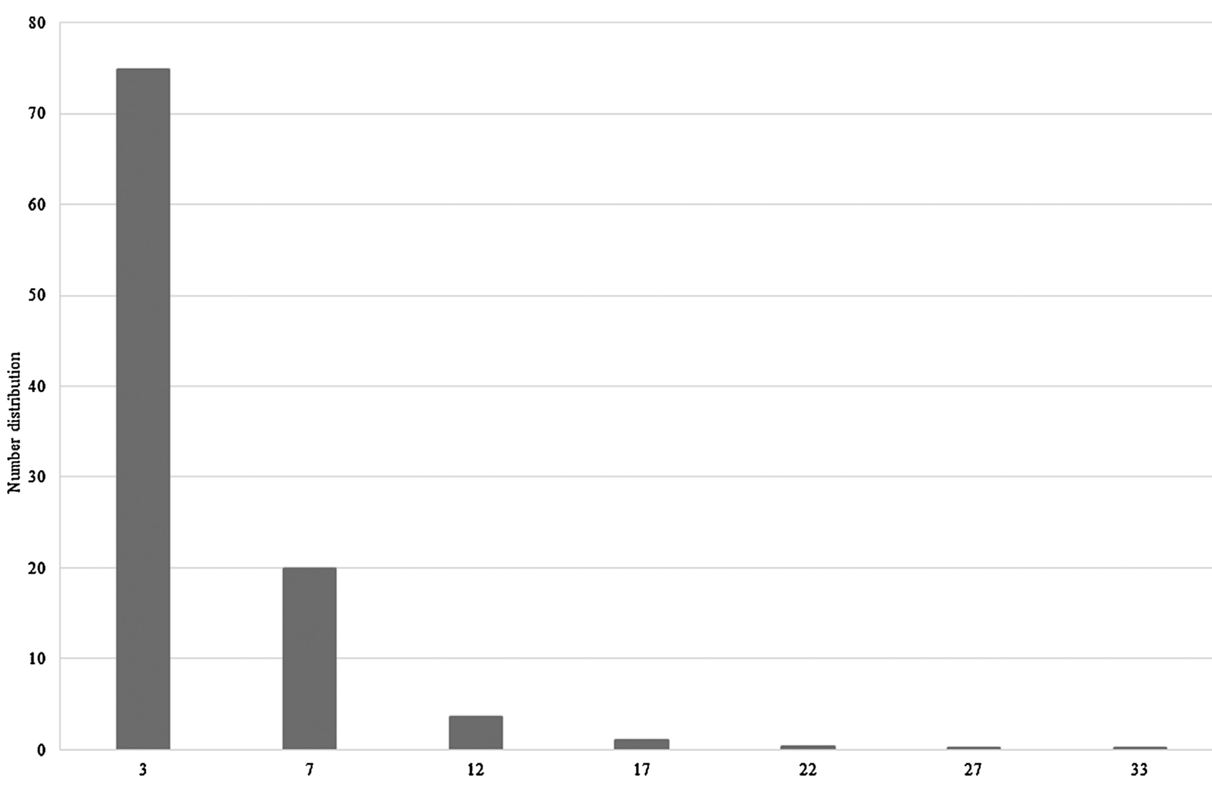


**Fig. 7.** Analysis of the particulate. Number distribution vs. $d_F$.

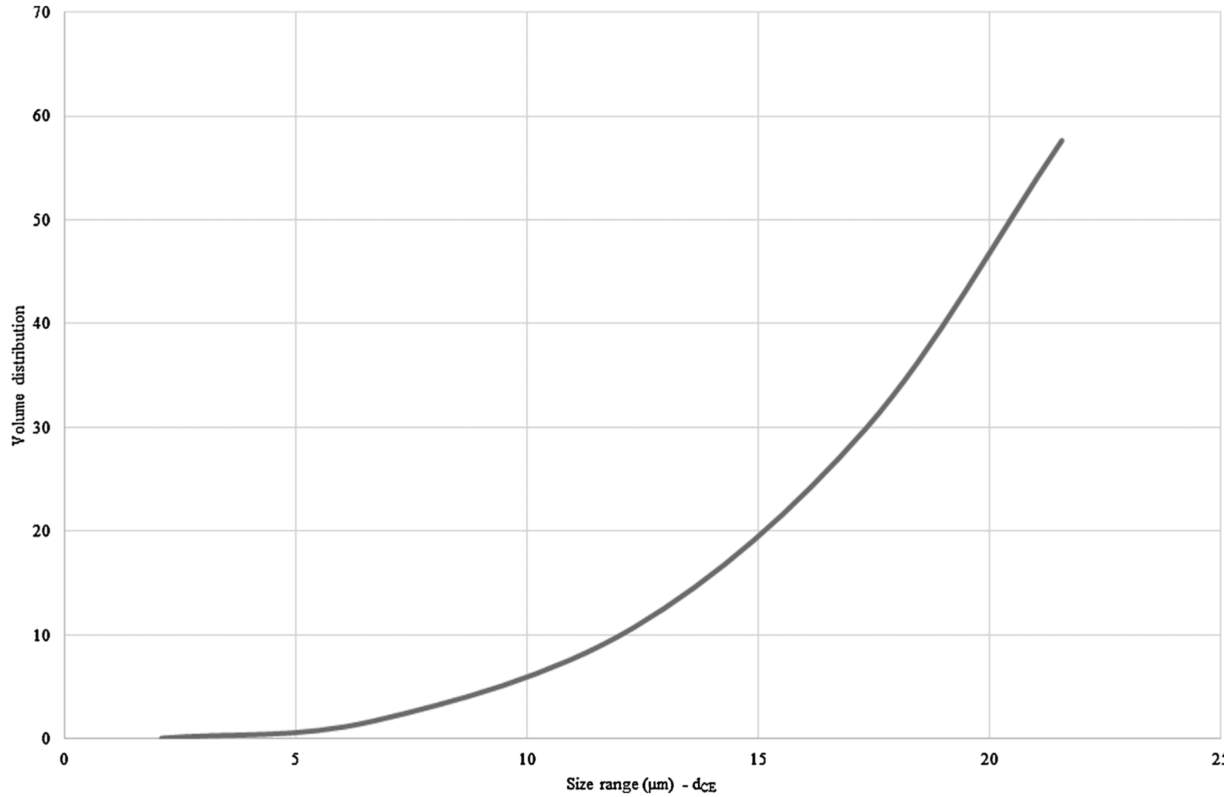


**Fig. 8.** Analysis of the particulate. Volume distribution vs. $d_{CF}$.

the results of the volume distribution of the particulate fraction of the GRP powder are shown. The volume of each particle has been calculated starting from the value of the $d_{CE}$ as the volume of the equivalent sphere.

As for regards the size distribution of the fibers, the results, based on a number distribution, are reported in Fig. 9. The results are shown representing the percentage of particles in each size group on the basis of the $d_F$ value.

In Fig. 10 the results of the volume distribution of the fibrous fraction of the GRP powder are shown. The volume of each particle has been calculated as a cylinder where the radius corresponds to the half of the minor axis of the fit ellipse and the height to the $d_F$.

The particle size distribution and the average particle size of the GRP powder have been also determined by laser diffraction particle sizing analysis (LD psa) and the results are reported in Figs. 11 and 12.

## 4. Discussion

Pictures of a GRP powder, coming directly from a shipyard as an industrial by-product, taken by electron microscope show different and complex shapes of particles (Figs. 2(a) and 13). In fact, different geometric expression could be observed such as cylinder, rectangular prism and sphere.

For the above reason, as far as the evaluation of particle size and size distribution is concerned, the use of an image analysis (IA) approach is proposed based on scanning electron microscopy (SEM) and ImageJ processing program.

As for regards the presentation of the results of the particle size analysis it is often convenient to report parameters based upon the maximum particle size for a given percentage volume of the sample. Percentiles are defined as $X_{aB}$ where:

- $X$ = parameter, usually $D$ for diameter;
- $a$ = distribution weighting, e.g. $n$ for number, $v$ for volume, $i$ for intensity;
- $B$ = percentage of sample below this particle size e.g. 50%, sometimes written as a decimal fraction i.e. 0.5.

For example, the $D_{v50}$ would be the maximum particle diameter below which 50% of the sample volume exists – also known as the median particle size by volume.

In order to highlight the inappropriateness in the use of indirect advanced analysis methods, such as those involving scattered or diffracted light or laser, for the analysis of complex shapes, the results obtained from LD psa have also been reported (Figs. 11 and 12). Such indirect methods assume the particle to be spherical, which is not the predominant case with GRP materials as can be seen from Figs. 2(a) and 13. In fact, laser diffraction measures particle size distributions by measuring the angular variation in intensity of light scattered as a laser beam passes through a dispersed particulate sample. Large particles scatter light at small angles relative to the laser beam and small particles scatter light at large angles, as illustrated below (Fig. 14).

The angular scattering intensity data are then analyzed to calculate the size of the particles responsible for creating the scattering pattern, using the Mie theory of light scattering. The particle size is then reported as a volume equivalent sphere diameter (Stojanovic and Markovic, 2012). As for regards the results of LD psa the most common percentiles usually reported are the $D_{v10}$, $D_{v50}$ and $D_{v90}$ extrapolated from the cumulative plot and reported in Table 1.

In order to compare the results coming from IA and LD psa, volume distributions have been reported for both the particulate fraction and the fibers of the GRP powder. As for regards the results of IA the calculated percentiles are reported in Table 2, for the particulate fraction, and Table 3 for the fibers.

It is possible to observe that the LD psa tends to underestimate the presence of the particulate fraction (overestimating the value

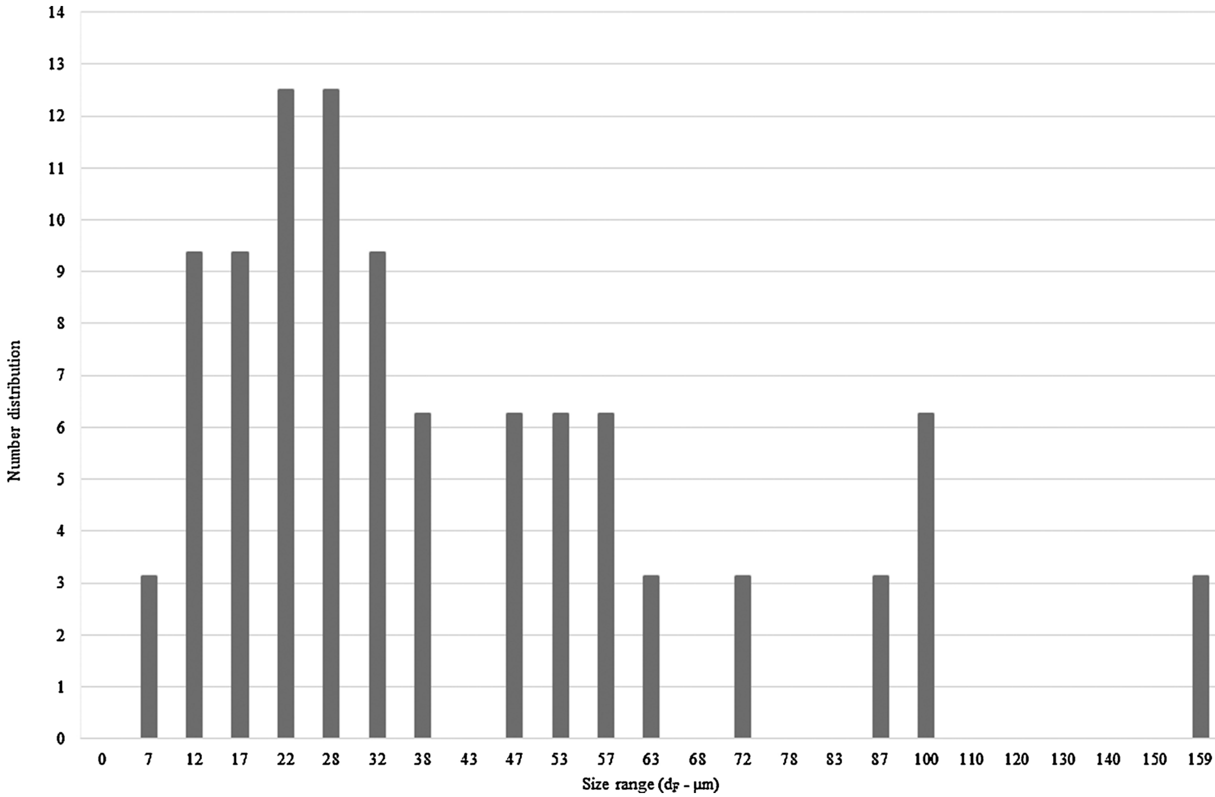


**Fig. 9.** Analysis of the fibers. Number distribution vs. $d_F$.

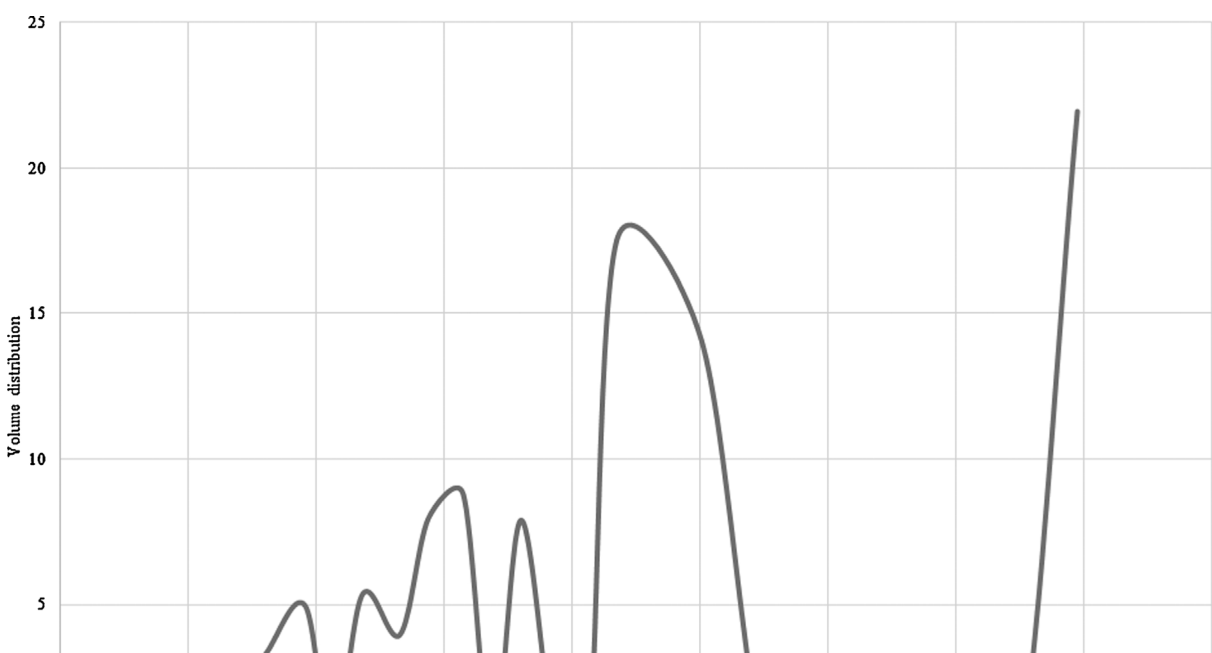


**Fig. 10.** Analysis of the fibers. Volume distribution vs. $d_F$.

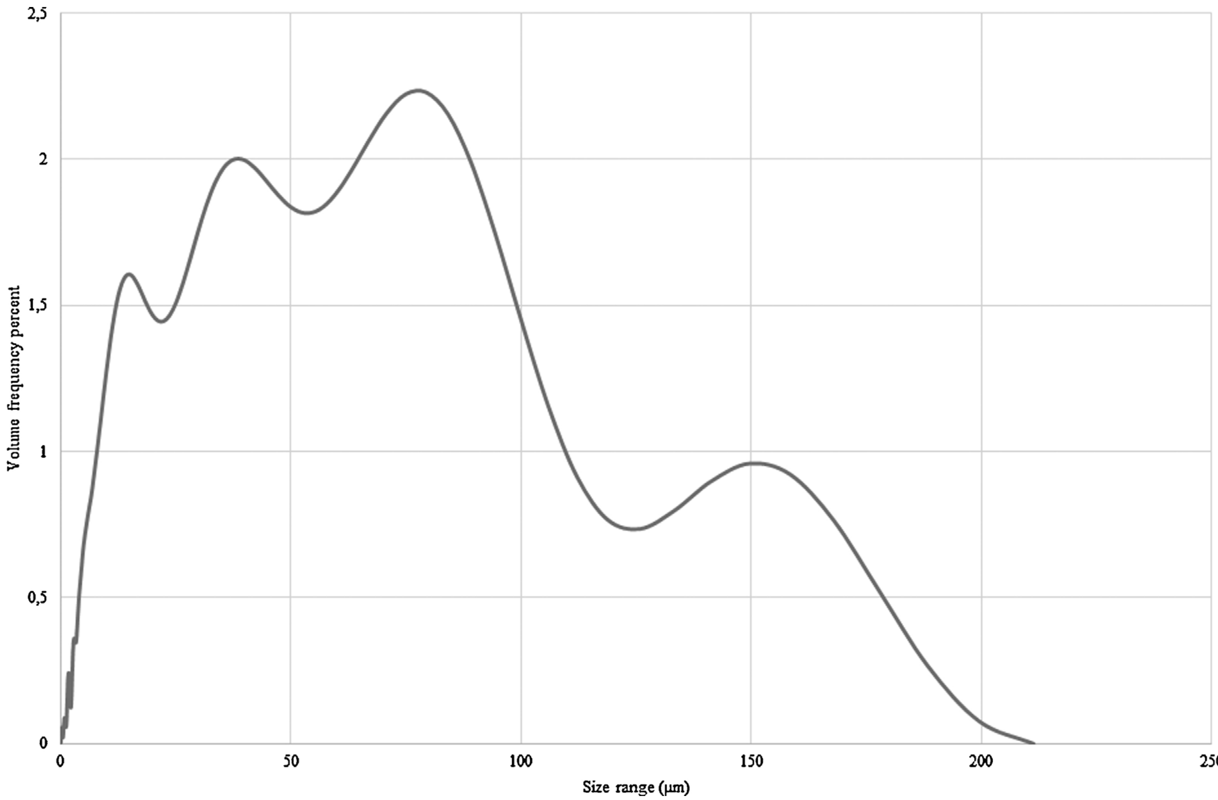


**Fig. 11.** Particle size distribution by laser diffraction.

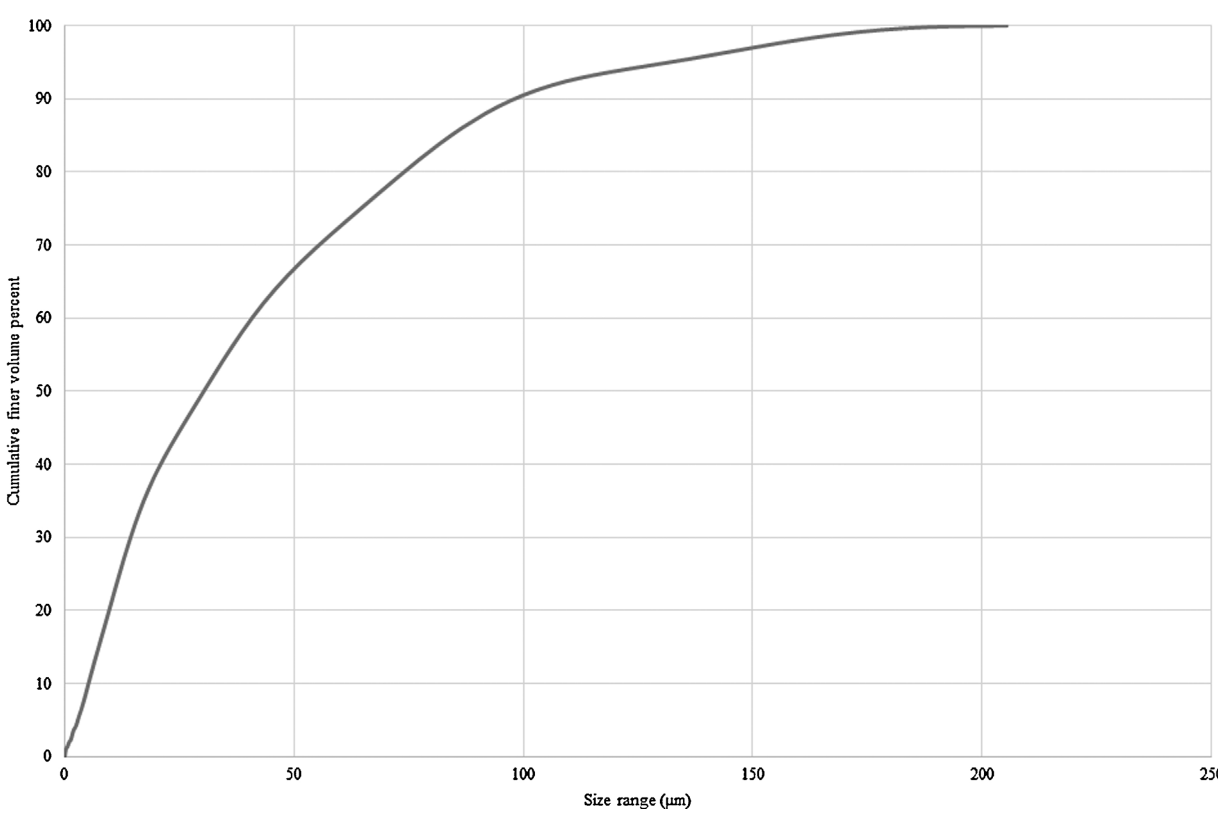


**Fig. 12.** Average particle size by laser diffraction.

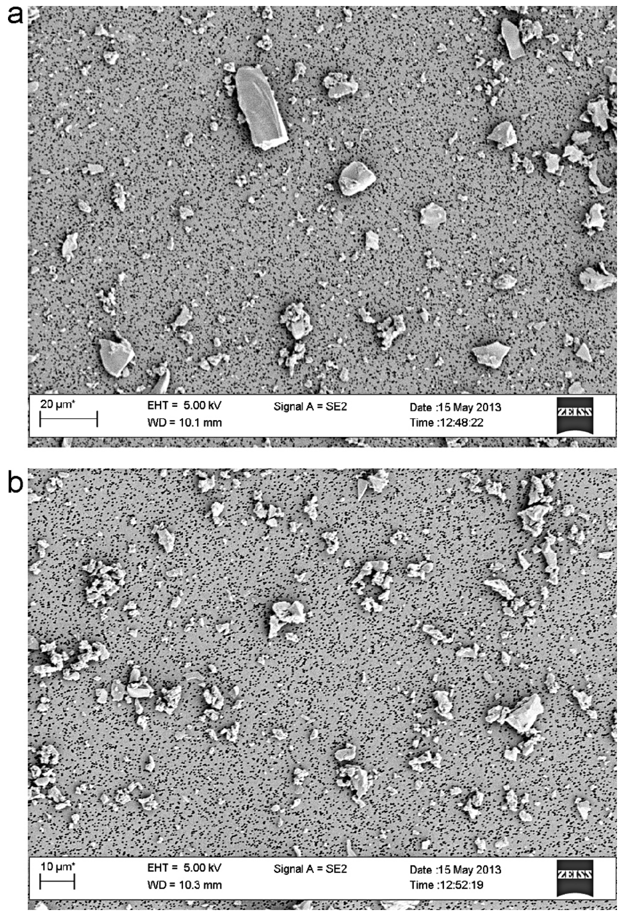


**Fig. 13.** Series of photomicrographs of GRP particles collected by electron microscopy.

of the mean diameter) when compared with the results obtained by IA method. The differences are probably due to the assumption about spherical particles in the LD psa method. In case of different and complex shapes of particles, IA methods seem to be the most accurate given that they directly observe and analyze the particles. However, they provide a bidimensional image, so errors may also be introduced by the unknown vertical dimensions, although they

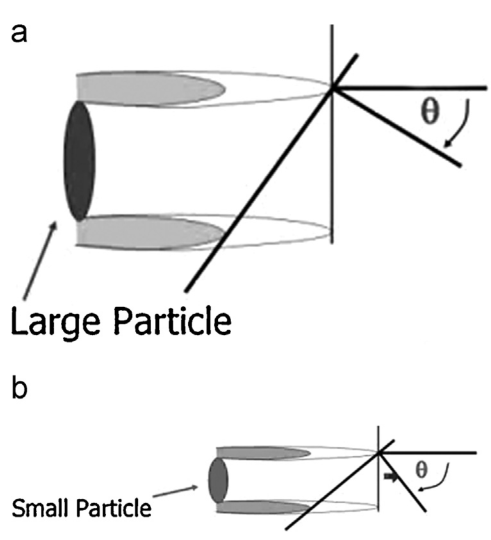


**Fig. 14.** Angular variation of light scattered as a laser beam passes through a large (a) and a small (b) particle.

**Table 1**
LD psa: $D_{v10}$, $D_{v50}$ and $D_{v90}$ extrapolated from the cumulative plot.

| | |
|---|---|
| $D_{v10}$ | 5 μm |
| $D_{v50}$ | 31 μm |
| $D_{v90}$ | 97 μm |

**Table 2**
IA: extrapolated percentiles relative to the particulate fraction.

| | |
|---|---|
| $D_{CEv40}$ | 17 μm |
| $D_{CEv100}$ | 22 μm |

**Table 3**
IA: extrapolated percentiles relative to the fibers.

| | |
|---|---|
| $D_{Fv10}$ | 38 μm |
| $D_{Fv50}$ | 83 μm |
| $D_{Fv100}$ | 159 μm |

are often considered very small in comparison to the horizontal dimension.

## 5. Conclusions

- Emerging sustainability issues compel a different from usual approach to solid waste and industrial by-products disposal and natural resources management, exploiting as much as possible the feasibility of reusing the former ones to replace the latter ones. This opportunity has already been proven in construction materials, where many industrial by-products are profitably employed as filler addition, provided that their chemical and physical nature is not deleterious to mechanical performance and that their grain size distribution fits in the material microstructure.
- GRP waste powder is a common by-product, consisting of polymer granules and glass fibers, whose use in cementitious products has recently been exploited, proving suitable as either cement replacement or filler addition to concrete mixtures depending on its fine grain size distribution. Its reutilization proved to be effective in lowering the cracking risk induced by restrained drying shrinkage, mainly attributable to the glass fibers volume fraction, and significantly lowering the capillary water absorption, mainly attributable to the polymer granules volume fraction. Therefore, the determination of these volume fractions as well as the corresponding particle size and distribution seems advisable and essential in order to optimize its use.
- An accurate evaluation of the mean diameter value as well as the maximum diameter of the particles is generally compromised by the particular morphology of the GRP waste powder, constituted by irregular polymer granules and glass fiber fragments. Usual size analysis techniques, as laser diffraction, being unable to distinguish the particles shape, introduce oversimplified approximation, which can be unacceptable, since the size distribution is often of critical importance to the way the material performs in use. Indeed, this technique tends to overestimate the value of the mean diameter and underestimate the finest particulate fraction.
- Therefore, the use of image analysis method, based on scanning electron microscopy (SEM) and ImageJ processing program, is proposed in order to evaluate the morphology of the particles and measure the particle size and size distribution of fine GRP waste powder. Such a method shows a great potential to be considered as a standardized method of measurement and analysis for the characterization of the grain size and size distribution of GRP particles as well as any other granular material containing fibers in order to optimize its recycling potential.

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