ISO 1183 – DETERMINATION OF PLASTICS DENSITY USING THE IMMERSION METHOD

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The Research Metrology and Certification Centre – Testing Laboratory is formed by a group of experienced employees dealing with the transfer of metrological knowledge and normative requirements for laboratory and industrial applications. In effect the testing methods based on mass measurements in micro and macro scale can be improved on a continuous basis.

INTRODUCTION

For several decades various plastics have been applied in key fields of the industry, starting from food industry and ending up with complicated medical applications. The products deriving from plastics can offer any functional features in terms of shape, aesthetics, mechanical parameters, temperature, and currently biodegradability too.

In retrospect, high popularity of plastics since the 1960s has been driven by a need to replace costly natural materials with inexpensive substitutes that proved acceptable in terms of quality. At present attention is paid not only to strong points but also negative aspects of using plastics, such as recycling issues and generation of micro and nano plastic. Sadly the 21st century is likely to be remembered as the plastics age. According to the OECD (Organisation for Economic Co-operation and Development), over 353 million tones of plastic waste were produced in 2019, and these numbers are bound to rise to around 1014 million tones by 2060. It must be noted that plastics do not degrade definitely but break down into smaller fractions, i.e. micro and nano particles, that spread uncontrollably throughout the globe. In fact it is not possible to eliminate plastics as they play an important role in our lives as products and raw materials. However, designing new solutions and inspecting their quality with a view to minimising their negative impact on the environment is something that can be done. In this respect, it is critical that numerous parameters of newly designed plastics, including their densities, are supervised.

There are a few reasons why verification of plastic density is crucial. Above all it is a guarantee that the material meets specific quality-related standards and remains consistent throughout the production batch. It is of paramount importance in B2B relationships, where the process-based approach in Quality Management Systems is applied. Mechanical properties of a ready-made product, such as durability, hardness, flexibility, can be closely associated with density of the granule used in the engineering process, especially when injection, extrusion and moulding procedures are not optimised. Plastic density is usually tested in accordance with requirements of such standards as ISO 1183:2019, ASTM D792:20, yet the OIML G 14:2011 document may also be applicable. Normative documents describe methods but fail to account for numerous issues that may emerge in the measurement process. Proper interpretation of results requires consideration of potential sources of errors, for example presence of air bubbles, sample heterogeneity, stability of the measuring system, etc. Care for details and testing diligence are critical for assurance of credible results. Typical density for some plastics have been showed in the table 1.

Table 1. Densities of some plastics

Name	Symbol	Density [g/cm3]
Acrylonitrile-butadiene-styrene	ABS	1.04 ÷ 1.06
Cellulose acetate	CA	1.25 ÷ 1.35
Polypropylene	PP	0.85 ÷ 0.92
High-density polyethylene	HDPE	0.89 ÷ 0.93
Low-density polyethylene	LDPE	0.94 ÷ 0.98
Polybutene-1	PB	0.91 ÷ 0.92
Polystyrene	PS	1.04 ÷ 1.08
Polyamide	PA	1.01 ÷ 1.09
Polyamide 6,6	PA 66	1.13 ÷ 1.16
Poly(methyl methacrylate)	PMMA	1.16 ÷ 1.20
Poly(vinyl acetate)	PVA	1.17 ÷ 1.20
Cellulose propionate	CP	1.18 ÷ 1.24
Polycarbonate	PC	1.20 ÷ 1.22
Poly(vinyl alcohol)	PVAL	1.21 ÷ 1.31
Poly(vinyl fluoride)	PVF	1.30 ÷ 1.40
Poly(ethylene terephthalate)	PET	1.38 ÷ 1.41
Poly(vinyl chloride)	PVC(-U)	1.38 ÷ 1.41
Polyoxymethylene	POM	1.41 ÷ 1.43
Polytetrafluoroethylene	PTFE	2.10 ÷ 2.30

Plastic density can be determined using a pycnometric method or gradient column method too. Still, regardless of the method in use, the measurement result must be precise.

RESEARCH METHOD

The immersion method involved the use of the AS 220.X2 PLUS balance with an elementary unit of d=0.1mg for mass measurement, and a special set of weighing pans that allowed measuring the sample mass in the air and liquid. The measurement principle adopted the Archimedes' law, according to which the body immersed in the liquid appears to lose as much weight as the weight of the liquid displaced by that body. That simple and universal technique allows testing samples of various shapes. This is why it proves versatile and useful in a number of applications. Determination of the plastic density using the immersion method rests on calculations as per the following equation:

$$\rho_s = \frac{M_1}{M_1 - M_2} x \rho_c \quad (1)$$

where: ρ_s – test sample density
M_1 – sample mass in the air
M_2 – sample mass in the liquid
ρ_c – liquid density

While measuring the mass of the body in the liquid, there are two forces: gravitational and buoyant. Therefore the dependency describing the sample (in liquid) mass measurement result takes the form showed in the equation (2).

$$R = F_G - F_W = (\rho_c \times V \times g) - (\rho_w \times V \times g) \quad (2)$$

where: R – measurement result
F_G – gravitational force
F_W – buoyant force
ρ_c – body density
V – volume of body or volume of displaced liquid is equal to volume of part of the body immersed in liquid
g – acceleration of gravity
ρ_w – liquid density

The characteristics of the immersion method with regard to density measurement has been showed in the figure 1. It is a default characteristics for all laboratory balances that are presently manufactured by Radwag.

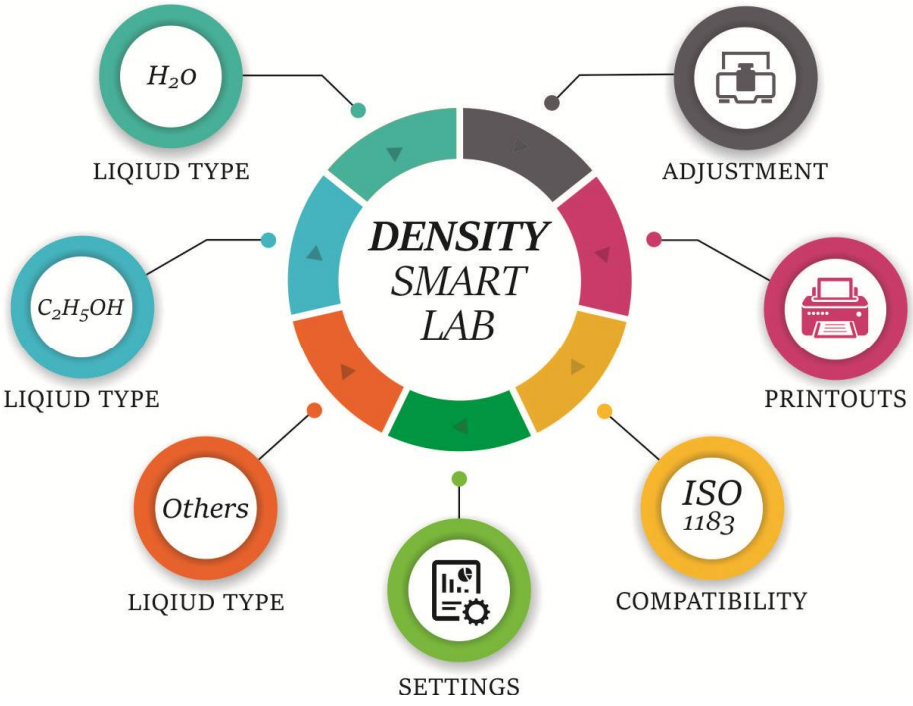

Figure 1. Solid density – method characteristics

Depending on density of the test sample, two scenarios are possible when the test takes place in the distilled water (ρ=1 g/cm^3). In the first scenario, the value of the gravitational force is higher than the buoyant force, as a result of which the sample gravitationally falls onto the lower weighing pan. In the second scenario, the buoyant force is higher than the gravitational force, and consequently the sample floats on the surface of the liquid. To perform the measurement, the sample must be positioned under the lower weighing pan, which requires the balance operator to be properly skilled. These differences has been presented in the figure 2.

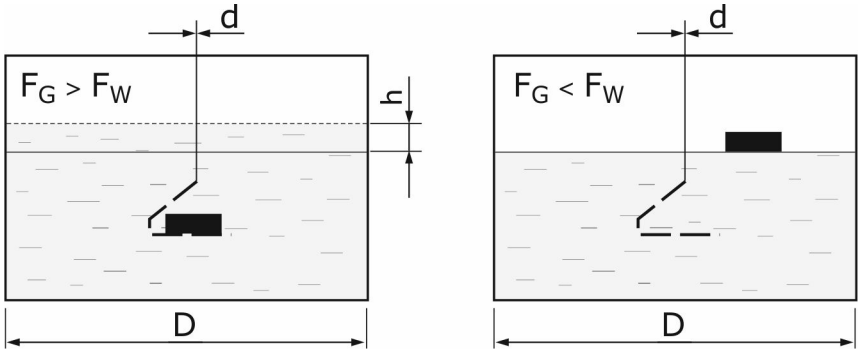

Figure 2. Buoyant force in testing plastics density

The immersion liquid is usually distilled water whose density is ca. 0,99g/cm$^{3.}$ It is also possible to use ethyl alcohol, density at 20^{o}C ~0,79g/cm^{3} , for which no sample floating effect is recorded for most plastics.

The factor that hinders sample mass measurement in the liquid is air bubbles that may adhere to greasy surfaces of the sample. Assuming the air bubble is a perfect sphere with a diameter of 1 mm, the equation (3) describes its diameter.

$$V_p = \frac{4}{3} \times \pi \times r_p^3 \tag{3}$$

where: V_P – air bubble volume
r_P – air bubble radius
π - 3,141592...

Considering the effect of gravitational force and buoyant force, it can be estimated that a single air bubble triggers the error of 0.01 g/cm3 in determining the sample density into the analysis. For this reason the visual inspection of the sample weighed in the liquid is required. Immersion of the sample in the liquid causes the part of the body to displace to the altitude (h), (fig. 2), and the liquid that is pushed upwards faces resistance formed by a string that connects weighing pans. For very precise analyses, the impact of this phenomenon can be eliminated through a correction factor that must be calculated from the equation (4). The value of this factor accounts for the thickness of the string (d) and beaker diameter (D) in which the test is performed.

$$WCC = 1 - 2 \times \frac{d^2}{D^2} \tag{4}$$

The conclusion that can be drawn from the equation (4) is that for the minimum thickness of the string on which the lower weighing pan is suspended and maximum diameter of the beaker, the correction factor WCC aims to reach the value of 1. For the set showed in the figure 3, the WCC value is 0,9985, and therefore proves insignificant.

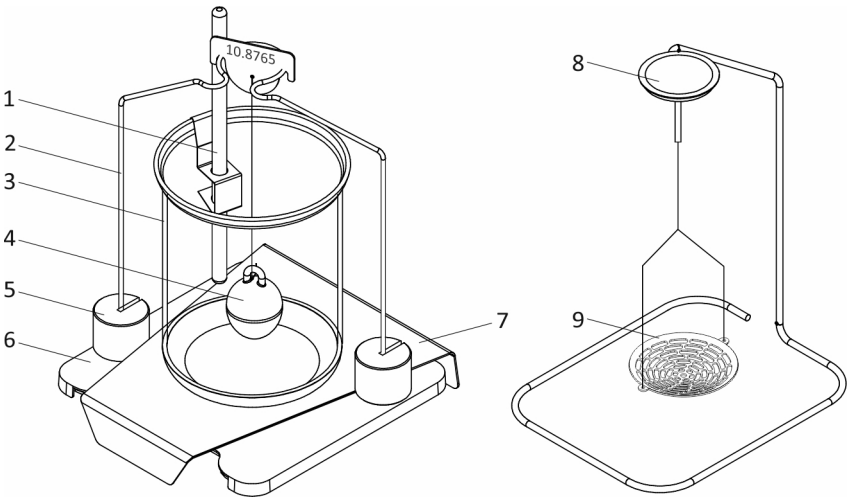

Figure 3. Solid and liquid density testing unit
1 – control thermometer, 2 – weighing pan rack, 3 – beaker, 4 – plunger, 5 – extra load, 6 – balance weighing pan, 7 – base, 8 – upper weighing pan, 9 – lower weighing pan

Although the immersion method is relative easy to adapt, there is a series of requirements that affect analysing accuracy:

- determination of plastics density using the immersion method must be performed in controlled climatic conditions in order to avoid changes to immersion liquid and test sample.

- the balance must not be positioned near doors, windows, air conditioner, fans, passageways or other places and air-blow producing devices as they may result in unstable indications and prolonged weighing time.

- the sample must be degreased, homogeneous, devoid of air bubbles and pollutants and surface defects that could affect the measuring accuracy.

- density measurements of the so-called recycled plastic granules may be subject to increased random error in view of potential sample heterogeneity that derives from secondary processing.

RESEARCH MATERIAL

The research material was composed of 13 types of plastic granules whose density was verified after the conditioning period using the solid density testing unit, manufactured by Radwag Wagi Elektroniczne. While testing, stable ambient conditions were provided, that is the temperature was 23^{o}C, and humidity 54%. The list of samples is showed in the table 2.

Table 2. List of plastic granule samples

Item	Name
1.	PA 6 GF 15 – Ravamid B GF 15 BK 45
2.	PA 66/6 FR 30 – Slovamid 66/6 FRC 3 TS 315/9M
3.	ABS – Polylac PA 717C
4.	PP – Borealis, homopolymer
5.	LDPE FT 3200
6.	MPE 1327 MD, ethylene and 1-hexene copolymer
7.	HDPE – HYA 600
8.	EVA FLOO 119, ethylene and vinyl acetate copolymer
9.	PC /ABS PULSE GX70 NATUR
10.	ASA LURAN S778 T SPF 30 SW 36831
11.	DAPLEN E E058AL-9557
12.	TPE-S BADAFLEX 60A 5123 FR S1
13.	PA6 SLOVAMID 6 OB 229 RED

Taking into consideration the conditions and circumstances as well as technical capabilities related to density inspection performed by plastic granule manufacturers and recipients, the samples were not initially melted using a plasticiser (at the first stage of the research). Attention must be however paid to the fact that determination of the MFI (Melt Flow Index) with the use of the plasticiser is one of essential pieces of information for the plastics processing technology. As part of specifying the MFI, continuous pieces of melted material are obtained and then possibly used for density testing. Such a procedure was adopted for the sample number 7 and 5 in order to declare whether material melting has a significant influence on its density.

RESEARCH SECTION

The purpose of the research was to compare the accuracy and precision of the method, depending on the type of immersion liquid, and to demonstrate the ergonomics as well as strong and weak points of research methods. The density results obtained while testing were compared to reference values from product data sheets. The plastic density difference with regard to the reference density may result from presence of additives in the granule and research method imperfections. The density results obtained during measurements in the distilled water and ethanol are showed in the table 3.

Table 3. Density of plastic granules, depending on the type of immersion liquid, mean ± S.

Sample		Granule density [g/cm^3]				
		Ref. data	H_2O	$\|\delta\|_{H2O}$	C_2H_5OH	$\|\delta\|_{C2H5OH}$
1.	PA 6 GF 15	1,23	1,20 ± 0,009	0,03	1,21 ± 0,008	0,02
2.	PA 66/6 FR 30	1,19	1,16 ± 0,005	0,03	1,17 ± 0,008	0,02
3.	ABS – Polylac	1,04	1,04 ± 0,007	0,00	1,04 ± 0,005	0,00
4.	PP – Borealis	0,89	0,90 ± 0,004	0,01	0,89 ± 0,001	0,00
5.	LDPE FT 3200	0,92	0,92 ± 0,002	0,00	0,92 ± 0,002	0,00
6.	MPE 1327 MD	0,93	0,92 ± 0,003	0,01	0,93 ± 0,001	0,00
7.	HPDE- HYA 600	0,95	0,92 ± 0,002	0,03	0,92 ± 0,001	0,03
8.	EVA FLOO 119	0,92	0,94 ± 0,004	0,02	0,94 ± 0,003	0,02
9.	PC /ABS PULSE GX70 NATUR	1,11	1,08 ± 0,001	0,03	1,07 ± 0,002	0,04
10.	ASA LURAN S778 T SPF 30 SW 36831	1,07	1,03 ± 0,003	0,04	1,02 ± 0,004	0,05
11.	DAPLEN E E058AL-9557	0,9÷1,0	0,97 ± 0,002	0,03	0,96 ± 0,001	0,04
12.	TPE-S BADAFLEX 60A 5123 FR S1	1,30	1,27 ± 0,005	0,03	1,26 ± 0,003	0,04
13.	PA6 SLOVAMID 6 OB 229 RED	1,14	1,12 ± 0,003	0,02	1,11 ± 0,003	0,03

The conclusion that can be drawn from the results is that testing of granule density may be performed alternatively in the distilled water or ethyl alcohol. The difference between the density results with regard to both methods is 0.01 g/cm^3 at the most. Importantly, measuring samples whose density is lower than 1g/cm^3 requires the operator to be more experienced and diligent.

To place the sample under the lower weighing pan, the operator needs to immerse the tweezers with the sample under the weighing pan and then to release the sample. As a result of buoyancy, the sample should be located under the lower weighing pan, thus causing the balance to change the indication. While this operation is performed, any shocks are undesired, and therefore the procedure must be collision-free. Measuring precision in the testing cycle expressed as a the standard deviation in the series of measurements ranged from 0.009 to 0.001g/cm^3 and was not dependent upon the type of test liquid.

From the metrological point of view, the value of random error should not be higher than 0.003g/cm^3. In case of such a value, one can be sure that the maximum impact of the random error on the measuring result will not exceed ca. ±0.0081g/cm^3. As for the worst precision obtained for the sample PA 6GF15, the measurement in water, the impact of random error on density result is now 0,0243g/cm^3, which may considerably misinform about the real granule density. The view of that sample in the test is showed in the table 4.

Figure 4. PA 6 GF 15 granule testing in distilled water
Measuring the sample mass in the air and liquid on the lower weighing pan

The above-stated conclusions may serve as a basis for improving research methods and inform the operator that the average value does not necessarily always represent the precise result. The terms related to precision of research methods can be accessed in the PN-ISO 5725-1 ,,Accuracy (correctness and precision) of measuring methods and measurement results, Part 1: general principles and definitions" document. Unfortunately the evaluation of the product quality at the output to the system or during engineering processes must be based on metrology, i.e. measurement science. At present numerous operators of diverse measuring systems are in quest of ready-made solutions in the field of research methods to obtain reliable and precise result in exchange for low personal and economic contribution. Difficulties emerge when product parameters are verified in B2B relationships and the test results differ.

The test results can be documented by means of traditional printout from the printer or computer application. Using the so-called non-standard printouts, documentation may be expanded to include further information fields.

----------Solids density----------	
Date	2024.12.30
Time	11:56:29
Balance S/N	654370
User	Alice
Liquid	Water
Temperature	22.0 °C
Liquids density	0.99780 g/cm3
Weighing in air	0.7381 g
Weighing in liquid	-0.0673 g
Density	0.914423 g/cm^3

Signature	

Figure 5. Solid density testing printout

While selecting the weighing system used for density measurements, it is necessary to consider its data-collecting capabilities. The most advanced system is the one equipped with the 5Y terminal that can record all density testing reports. Additionally it is supplied with a series of ergonomic safety functions, such as system logging and the so-called Digital Weighing Auditor metrology, fig. 6.

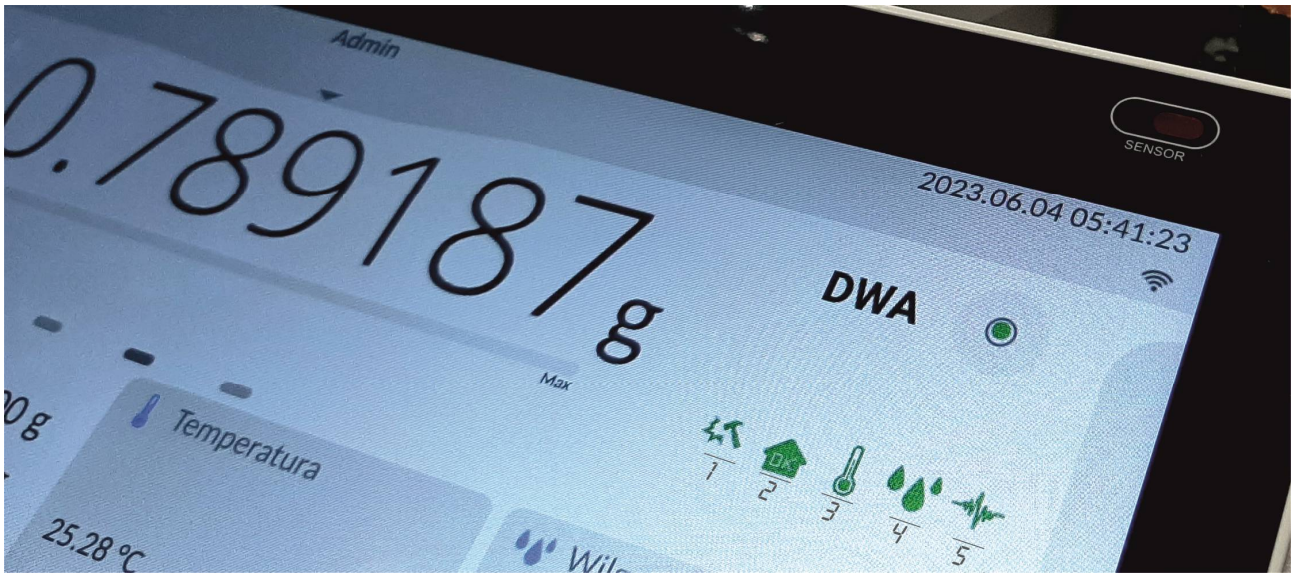

Figure 6. Part of 5Y balance screen – Digital Weighing Auditor application
1 – weighing quality monitor (shocks), 2 – Digital Weighing Auditor status, 3 – temperature control, 4 – humidity control, 5 – surface vibration detection

SUMMARY

The accuracy of determination of plastics density using the immersion method is dependent upon numerous factors, such as sample properties, working environment stability, operator's skills, presence of air bubbles, preparation of the sample for testing, etc. There is no doubt that the best situation is when the granule sample is melted with the use of the plasticiser into uniform pieces. In such a case the size of the test sample may be at least 1g, as required by ISO 1185 section. 5.1.3.

It is highly important because the influence of random error on the average density value would be negligible. The reality however is that plastics processing plants rarely determine the MFI as it is assumed that it is a permanent value for a specific material. The research has demonstrated that accurate testing of density is feasible also for a considerably smaller samples, yet proves more time-consuming and requires diligence in the testing process.

References:

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