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Artigo

# Difference Between the Results Obtained by DIN and ASTM Procedures in the Bulk Density of Solid Biomasses

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## Diferença entre os Resultados Obtidos pelos Procedimentos das Normas DIN e ASTM na Densidade a Granel de Biomassas Sólidas

**Resumo:** A padronização dos testes garante que a metodologia utilizada tenha uma reprodutibilidade do experimento e uma confiabilidade do resultado. No entanto, para realizar alguns testes em escala de laboratório, é necessária uma adaptação para ajustar a uma quantidade de amostra ou a um parâmetro não operacional. O objetivo deste trabalho foi validar e comparar dois testes diferentes de densidade aparente para biomassa, seguindo as normas ASTM E873 e DIN EN 17828, mas com alterações (escala laboratorial) no volume a ser medido da amostra e do recipiente. recipiente foi feito com dimensões reduzidas proporcionalmente. O procedimento foi realizado por 6 operadores (3 DIN e 3 ASTM) com seis materiais (biomassas) de diferentes características físicas. Os resultados mostraram que os procedimentos de ambos os padrões resultam em valores aproximados em relação à densidade aparente dos materiais. Também foi observado que a heterogeneidade do material e os procedimentos manuais (sugeridos pela norma) contribuíram para algumas divergências nos resultados. Por fim, concluiu-se que uma repetição maior é recomendada para reduzir o erro devido à variação dos resultados entre as medidas.

***Palavras-chave:*** Método analítico; biocombustível sólido; reprodutibilidade; mensurações.

## Abstract

The standardization of the tests ensures that the methodology used guarantees a reproducibility of the experiment and a reliability of the result. However, to perform some tests on a laboratory scale, an adaptation is necessary to adjust to a quantity of sample or to a non-operational parameter. The objective of this work was to validate and compare two different tests of apparent density for biomass, following the standards ASTM E873 and DIN EN 17828, but with changes (laboratory scale) in the volume to be measured of the sample and the container. The container was made with reduced dimensions proportionately. The procedure was performed by 6 operators (3 DIN and 3 ASTM) with six materials (biomass) of different physical characteristics. The results showed that the procedures of both standards result in approximate values in relation to the bulk density of the materials. It was also observed that the heterogeneity of the material and the manual procedures (suggested by the standard) contributed to some divergences in the results. Finally, it was concluded that a greater repetition is recommended to reduce the error due to variation of results between as measurements.

***Keywords:*** Analytical method; solid biofuel; repeatability; measures.

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# Difference between the results obtained by DIN and ASTM procedures in the bulk density of solid biomasses

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## 1. Introduction

Laboratory tests are of paramount importance for the analysis and physical-chemical characterization of materials.[1-3] Standardization entities normalize some assays to ensure that the methodology used provides the reproducibility of the experiment and the reliability of the results.[4]

Two of the major institutions that regulate these procedures are: the American Society for Testing and Materials (ASTM) which is a US standardization body and the German Institute for Standardization, Deutsches Institut für Normung (DIN), which is an organ of Germany representing the International Organization for Standardization (wISO).

For the materials that compose solid fuels and biomasses, one of the important characteristics to be analyzed is the moisture content and the density,[5-8] mainly to estimate the transport and storage of the materials.[9,10] However, in order to perform some laboratory-scale analysis, in which the amount of material required by the test procedure is often not achieved, adaptations must be taken to suit the quantity of sample or any parameter not attainable.

In addition to checking the procedure for measuring material characteristics is important to evaluate the reliability of the methodology used. This verification can be done with the repeatability and reproducibility analysis of the method.[4]

Bulk density is a volume parameter used for solid biofuels, facilitating the estimation of the space required for the transportation and storage of these materials,[11] in addition, the energy density can be determined with other physical-chemical characteristics of the

material.[12] ASTM E873 and DIN ISO 17828 standards for bulk density determination limit the particle size of the material according to the size of the measuring vessel and can be used for grains, pellets or materials, generally from biomass sources, which have been subjected to processing and had a change in the particle size of the material.

The objective of this study was to compare the bulk density test procedure, following the recommendations of ASTM E873 (ASTM) and DIN ISO 17828 (DIN),[4,13] but with adaptations in the sample volume and the container test, due to the possibility of analyzing smaller quantities of material commonly received or processed in the laboratory scale.

## 2.Materials and Methods

### 2.1. Materials

Six biomasses with different physical characteristics (different particle sizes) were chosen in order to cover the high diversity of biomass used for bioenergy. The materials used for the analysis were: rice husk (sample 1), pinus sawdust (sample 2), guapuruvu sawdust (sample 3), eucalyptus bark sawdust (sample 4), eucalyptus sawdust (sample 5) and cotton linter (sample 6), as shown in Figure 1.

All the materials were oven dried and pre-processed (crushed or ground) and had their moisture content verified after the test based to the standard ASTM D3172-13.[14]

### 2.2. Container manufacture

Biomass bulk density were determined using a measuring vessel adjusted to a reduced size as described by ASTM E873 and DIN ISO 17828 in order to adapt to a smaller sample volume. Deviations from the dimensions of the test vessel are described in the methodologies. The height-diameter ratio was maintained.

The first procedure was DIN ISO 17828 - Solid Biofuels - Determination of Bulk Density (2013). Two cylindrical containers of transparent acrylic with different sizes and volumes were made. A larger one with a volume of 900 $cm^3$ (0.9 L) and a smaller one with a volume of 200 $cm^3$ (0.2 L) keeping the height-diameter-ratio dimensions from 1.25 to 1.50 as shown in figure 2a.

The second one was performed based on ASTM E873 - Standard Test Method for Bulk Density of Densified Particulate Biomass Fuels (2013). This standard defines a cubic container with dimensions of 305 mm of each side. A proportionally smaller container with reduced volume was used. The final dimensions of the container were 100 x 100 x 100 mm, totalizing a volume of 1,000 $cm^3$ (1,0 L), according to Figure 2b.

### 2.3. Test procedure

The determination of the density of each material was carried out in duplicate by 3 people (operator) for each standard (3 used the DIN standard and 3 used the ASTM standard). The methodology according to the DIN standard was carried out (by 3 operators) at the Laboratory


**Figure 1.** Samples: (1) rice husk, (2) pinus sawdust, (3) guapuruvu, (4) eucalyptus bark, (5) eucalyptus sawdust, (6) cotton linter

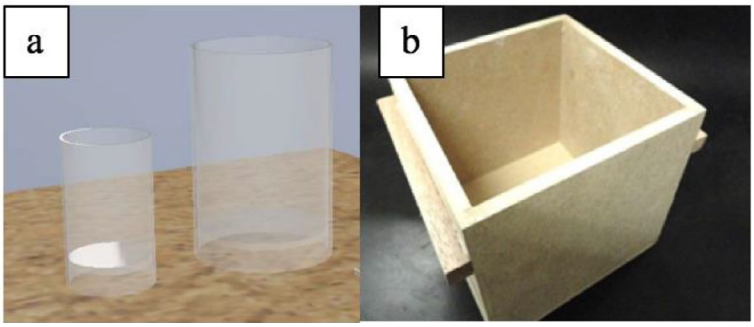


**Figure 2.** a) Containers for bulk density test (0.2 L and 0.9 L) constructed according to DIN ISO 17828. b) Container for bulk density test, adapted from ASTM E873

of Fuels and Lubricants of the Institute for Technological Research (IPT). The methodology according to the ASTM standard was carried out (by 3 operators) at the Biomass and Bioenergy Laboratory of UFSCar Sorocaba. The bulk density of the material was calculated using equation 1.

$$BD = \frac{m}{v} \quad (1)$$

where: BD = Bulk Density, in kg.m$^{-3}$; m = mass in g; v = volume in L.

The validation of the method was done by comparing the results of the three people (Op1, Op2 and Op3), showing the repeatability and reproducibility of the results, as well as their standard deviation. It should be noted that the ASTM standard does not define a reference value of repeatability, therefore the same reference value was considered for both methodologies. The reference value of repeatability and reproducibility is defined as follows:

For materials with a mean density of less than 300 kg.m$^{-3}$: the repeatability value should be below 2% and the reproducibility should be below 4%.

For materials with a mean density greater than 300 kg.m$^{-3}$: the repeatability value should be below 3% and the reproducibility should be below 6%.

The repeatability was made by comparing the value between the two checks of each operator. The results were also submitted to basic statistical analysis and analysis of t-test.

## 3. Results

The analyzed samples presented the moisture content (after drying) values of 6.3, 6.9, 6.5, 4.2, 5.4 and 10.8% respectively for samples one to six. The results of the analysis of the densities of the materials, following the methodology described by the DIN standard, are arranged in Figure 3.

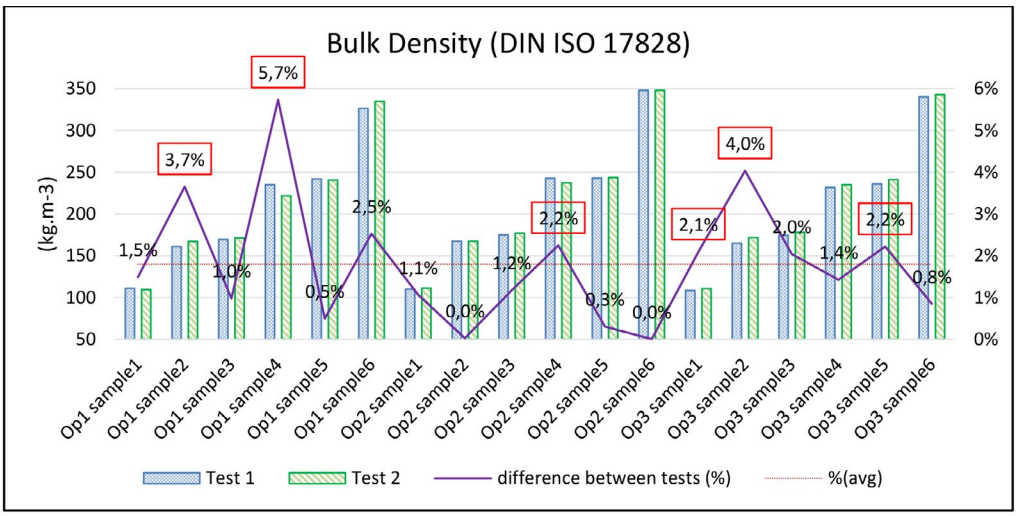


**Figure 3.** Measurements of bulk densities and percentage of variation between measurements, based on DIN ISO 17828

Figure 3 shows a bar graph with the duplicate measurements of the six sample types for each of the three operators and the difference in percentage measure between the first and second checks in the line graph. The percentage difference between the measures accepted by the standards is <2% (BD <300 kg m$^3$) and <3% (BD >300 kg m$^3$). The results showed that values above the standards occurred for all operators and all samples. For operator 1 the sample 2 (3.7%) and 4 (5.7%); operator 2 sample 4 (2.2%) and operator 3 sample 1 (2.1%), 2 (4.0%) and 5 (2.2%). It was possible to verify that the error occurred in a random way, it is not due to operator or some sample.

The results can be explained by the heterogeneity of the samples and, mainly, by technical procedures that depend on the ability of the operator, described by both standards.

Figure 4 presents the results of bulk density (by each operator) using ASTM standard.

Figure 4 showed that the apparent densities mesured according to the recommendations of the ASTM standard had a behavior similar to the previous results (DIN). The random variations observed using the DIN standard, also occurred using the ASTM standard. It should be noted that the ASTM standard does not establish repeatability limits, therefore the same values applied in the DIN standard were considered: <2% (BD <300 kg m$^3$) and <3% (BD >300 kg m$^3$).

The operators used for ASTM were not the same ones that performed the DIN bulk density. The operator 1 showed values above the standard for sample 3 (2.2%); operator 2 for samples 4 (2.5%), 5 (4.6%) and 6 (3.7%). The operator 3 presented results within the established limits. It was possible to verify that the error occurred in a random way, it is not due to operator or some sample.

Comparing the results of the average variation (dotted line) between all materials and operators in Figures 3 and 4, it was observed that the methodology recommended by the ASTM standard had a lower variation (1.58%) than the DIN procedure (1.80%). Perhaps the best explanation is in the fact of higher repetition of shocks, five for the ASTM standard and three for the DIN, which can lead to better accommodation of the particles inside the box. This characteristic of accommodation or compaction of the sample can be visually observed in the lower region of the container after submission of the same to the free fall shock, according to Figure 5.

The Table 1 shows the mean values of the measurements of all operators for each methodology.

It was possible to observe that sample 6 presented a high standard deviation for both methodologies used. Probably due to the heterogeneous physical characteristics of the cotton linter sample, which had the largest

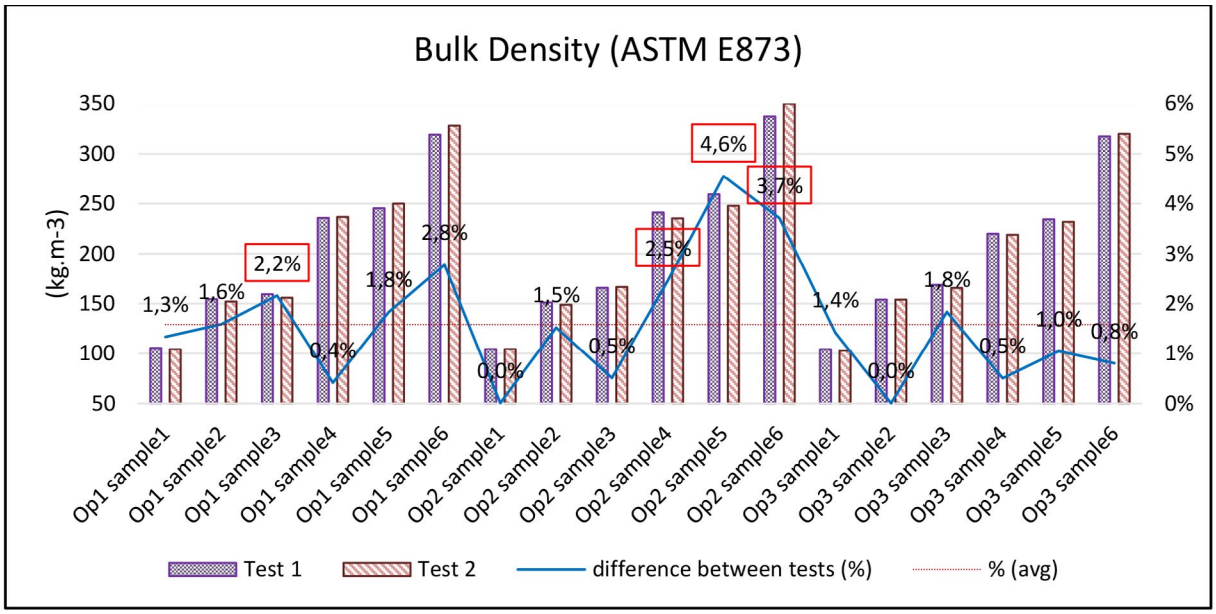


**Figure 4.** Measurements of bulk densities, based to ASTM standard, and percentage of variations between measurements

**Figure 5.** Container with rice husk sample (sample 1), highlighting the visually more compacted region due to the impacts suffered during the procedure

**Table 1.** Bulk density means and standard deviation

| | Sample | Bulk Density (kg.m$^{-3}$) | Standard Deviation |
|---|---|---|---|
| DIN ISO 17828 | Sample 1 | 110,26 | 0,39 |
| | Sample 2 | 166,71 | 1,86 |
| | Sample 3 | 174,24 | 2,69 |
| | Sample 4 | 234,08 | 4,75 |
| | Sample 5 | 241,06 | 1,96 |
| | Sample 6 | 339,96 | 7,17 |
| ASTM E873 | Sample 1 | 104,30 | 0,59 |
| | Sample 2 | 152,31 | 1,73 |
| | Sample 3 | 163,61 | 4,27 |
| | Sample 4 | 231,34 | 8,45 |
| | Sample 5 | 244,79 | 8,60 |
| | Sample 6 | 328,59 | 10,79 |

Source: Author (2017)

particle size. Also, cotton linter is a residue with seeds and traces of cotton (fibers) that remain together the seed. The fibrous material presented greater difficulty in accommodating the volume inside the container.

Another detail observed was the tendency of increase in the standard deviation, that is, greater probability of errors for samples with higher densities for both methodologies used.

Figure 6 shows the comparison of the mean values for the two methodologies.

The Figure 6 showed the difference (t-test) of results between the methodologies for sample 2 and for sample 3. The sample 6 presented a high standard deviation in both methods and statistically did not present significant differences (t-test) between DIN and ASTM.

## 4. Conclusion

It was concluded that both standards have similarities in their procedures and it was corroborated with the similarity between the mean values of density.

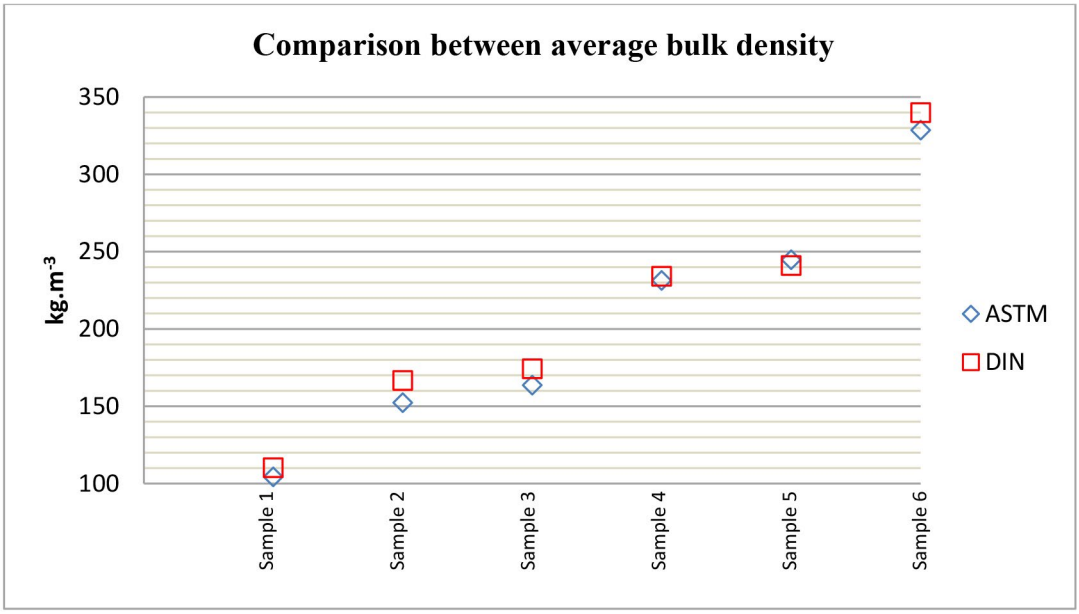


**Figure 6.** Comparison between the mean values of density values for both methods used

Both standards may generate errors in measurements depending on the physical characteristics of the samples being analyzed.

The physical characteristics of the sample and its density may influence the measurement, so it is suggested that the number of minimum repetitions should be increased, deviating from the recommendations.

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