Microwave aquametry according to ASTM D4643-17 for moisture determination in iron ore tailings

Aquametria de microondas de acordo com a ASTM D4643-17 para determinação da humidade em rejeitos de minério de ferro

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ABSTRACT
The gradual increase in the exploitation of iron ore leads to a need for the disposal of tailings from its processing. One of the key features of a granular system stacked is its moisture content. This work aimed at evaluating the use of microwave oven to determine the moisture content (following the ASTM D4643-17 standard) of iron ore dewatered tailings, for fast assessment of the geotechnical condition of every stacked layer on disposal areas. The preconized method has showed good agreement with the conventional oven method (standard assay), resulting in linear equation with coefficient of determination of 0.9953. However, the recommended method has shown less accuracy in the range of low moisture values (below 5 %), which does not represent a critical disadvantage from the point of view of geotechnical safety. The economic impact of the method advocated here will be great, since the movement of large volumes of tailings in limited areas leads to customary downtime, often resulting from waiting for confirmatory analytical results to practice within the safety limits.

Keywords: Moisture, Humidity, Iron Ore, Drying, Metrology.

RESUMO
O aumento gradativo da explotação do minério de ferro leva à necessidade de destinação dos rejeitos de seu beneficiamento. Uma das principais características de um sistema granular empilhado é seu teor de umidade. Este trabalho teve como objetivo avaliar a utilização de forno de micro-ondas para determinação da umidade (seguindo a norma ASTM D4643-17) de rejeitos desaguados (filtrados) de minério de ferro, para avaliação rápida da condição geotécnica de cada camada empilhada em áreas de disposição. O método preconizado apresentou boa concordância com o método de estufa convencional (ensaio padrão), resultando equação linear com coeficiente de determinação de 0,9953. Porém, o método recomendado apresentou menor acurácia na faixa de valores de umidade baixos (abaixo de 5 %), o que não representa uma desvantagem crítica do ponto de vista da segurança geotécnica. O impacto econômico do método aqui preconizado será grande, uma vez que a movimentação de grandes volumes de rejeitos em áreas limitadas leva a paradas habituais, muitas vezes resultantes da espera por resultados analíticos confirmatórios para se garantir a operação dentro dos limites de segurança.

Palavras-chave: Umidade, Minério De Ferro, Secagem, Metrologia.

1 INTRODUCTION

In mining activities usually large volumes and masses of materials are exploited. Several factors influence the amount of waste generated, including the ore extraction process, the location of deposits in depth, and the techniques available at the extraction sites, among other factors.

Two types of waste are intrinsically related to mining, tailings and waste rock. Waste rock is the material that comes from the overburden and sterile enclosing rocks. The tailings, in turn, result from ore dressing operations.

One of the most important steps in the tailings stacking process is the bulk material compaction, which aims to reduce the volume of voids, leading to a density increase of the compacted material, increasing its strength and load capacity. Other relevant features of the compaction process are the reduction of material permeability and the reduction of deformability.

The PDER Ipoema-Borrachudo Project is based on the disposal in a shared pile of waste rocks and tailings cakes, at the Mining Complex of Itabira (in Minas Gerais State, Brazil), by the Vale S. A. company. It is currently in an assisted operation phase. This project was motivated by the gradual increase in the production of iron ore concentrates and, consequently, the need for disposal of tailings from its processing.

The operational cycle for the disposal of dewatered tailings includes, among other steps, topographic survey, material spreading and ground levelling (rough grading), moisture determination and control of the degree of compaction.

In a review on aquametry, that is, the quantification of water content in solids and bulk materials, Pyper (1985) has categorized the water present in solids structurally: free water (or water bound to substrates by physisorption) and combined water. Free water is what is usually referred to as hygroscopic moisture. Combined water, in turn, can be:

- coordination water: when water molecules are directly linked to cations from the crystalline lattice (example: $MgCl_2$. 6 H_2O);
- network water: when water is not directly bound, but forms hydrogen bonds with other water species (example: one of the five water molecules from $CuSO_4$ .5 H_2O);
- hydronium ions: water is present in the crystal as $H30^+$ ions (as in the case of uranophane: Ca (H_30) 2(UO_2), $(Si0_4)_2$. 3H_2O);
- decomposition water: the generation of water occurs by thermodecomposition of hydroxides (for example: $Ca(OH_2)$);
- zeolite water: in this case, heating leads to water evolution at the crystalline lattice level, without chemical decomposition of the crystal itself (typically zeolite, known as molecular sieves).

Testing soil natural moisture is a routine in geotechnical laboratories around the world. The main method for this essay is oven drying, which uses a controlled temperature between 105 ºC and 110 ºC. Despite being a simple, accurate and reliable method, its main disadvantage is the need for drying time, ranging from 16 to 24 hours. In Brazil, this test is standardized by *NBR 6457/2016: Soil samples — Preparation for compaction tests and characterization tests*.

The time spent in these tests, within the scope of the PDER Ipoema-Borrachudo Project, significantly impacts the operational cycle, and may generate, in a future scenario, production interruptions, resulting from the lack of availability of areas for temporary disposal of tailings. Thus, more agile and efficient methods of moisture testing have been sought. One of them is the heating by "dielectric resonance" employing electromagnetic microwave (with wavelength, λ, in the range of 1.0 mm to 1.0 m), since the water molecule is highly polar (dipole due to oxygen with a negative electron cloud opposing the two positively charged hydrogen atoms). Due to the water polarizability, these dipoles oscillate in phase with the radiation, resulting in system heating.

It should be noted that this is not a "mechanical" resonance, in the classical sense, because the fundamental frequencies of water molecules, expressed by their vibrational spectrum, are above 1.0 THz (especially in the infrared range), being much higher than the operating frequencies of microwave ovens (see, for example, the spectra showed by Zhukova1 et al., 2014). In order to verify this bond resonance, in addition to considering the fundamental frequencies linked to intramolecular vibrational modes, one should also consider their intermolecular interactions (hydrogen bridges) when dealing with liquid water. As recorded by Vollmer (2004), by international convention (even to avoid interference with communication systems) residential microwave ovens operate in the 2.45 GHz band (equivalent to λ = 0.122 m). An interesting development of the theoretical basis of microwave aquametry, as well as the citation of several examples of its application in Brazil, is given by Severo (2016).

Kraszewski (1991) lists the following advantages, in principle, of microwave methods:

- the material ionic conductivity can be disregarded at high frequency (its effect is much smaller than with radio waves);
- physical contact between the equipment and the tested material is not necessary, which enables continuous and remote online measurements;
- the test is, in principle, non-destructive.
- microwave radiation propagates through opaque non-metallic media, in contrast to infrared radiation, which is absorbed near the surface.
- microwave methods are fast and safe, unlike ionizing radiation methods;
- microwave radiation is relatively insensitive to dust and water vapor, in contrast to infrared radiation.

Different researches have shown that there is a potential to use the microwave oven drying method to obtain fast and accurate moisture results for different soil types (e.g., Usmen and Kheng, 1986; Diprose, 2001; Tavares et al., 2008; Jalilian et al. 2017), including organic soils (Kramarenko et al., 2016a, b; Mohamad et al., 2020), in addition to fresh concrete (Nagi and Whiting, 1994) and aggregates (Mafuma and Muller, 2020).

However, procedures for using a microwave oven for moisture testing are not yet standardized in most countries. Notable among the countries that have this method standardized are the United States of America (ASTM D4643-00; ASTM D4643-08; ASTM D4643-17), Australia (AS 1289.2.1.4-2015; AS 1289.0: 2014) and France (NF P

94-049-1), as pointed out by Jastrzębska (2019). Nowhere, however, is there specific parameterization for filtered (dewatered) tailings from iron ore beneficiation.

The results obtained in different researches, for soils and cements, indicate that a short drying period (about 10 minutes) in a 600 watts microwave oven could effectively replace oven drying in 105 °C for 24 hours (Jalilian et al. 2017), representing a considerable advantage when there is a need for quick results (Gaspard, 2002). On the other hand, the microwave oven can promote excessive drying of soils (with loss of crystallization water) resulting in overestimated moisture values (Berney et al., 2011). That is the main objection raised against the use of microwaving. In this sense, such a method is not usually recommended for soils with high levels of heat-decomposable minerals such as halloysite, montmorillonite, gypsum and other hydrated materials, as highlighted by the laboratory lesson guide from the School of Engineering of São Carlos (Department of Geotechnics, 2021).

In this context, this article aims to analyze the feasibility of using a microwave oven to determine the moisture content of iron ore tailings samples resulting from the filtration process in the so-called Iron Quadrangle geological province, in Minas Gerais.

2 MATERIALS AND METHODS

The granulometric characterization of mining tailings used in this study aimed to classify the particles that constitute the soil according to their different sizes. In turn, knowledge of densities is important for the determination of several physical indices of a soil, such as the void index, and for tests involving sedimentation.

For the analysis of moisture content, 20 samples of iron ore tailings collected at different locations of the pile under study were used, half of which came from a road drain (BR, numbered from 1 to 10) that uses this material as a base, and the other half from a temporary deposit (DT), numbered from 11 to 20, both located in the Itabira Mining Complex.

It is noteworthy that each sample originated six subsamples, which were placed in metal capsules (three of them to be analyzed using an electrical oven and the other three using the microwave oven), except for the DT-18 sample that was divided into four capsules.

An Electrolux ME044 microwave oven (with a rated power of 1.3 kW) was used at full power to dry from 75 grams to 90 grams of total tailings. The study was carried out in order to determine the ultimate test time that did not allow the temperature to exceed

110 ºC, to avoid sample overheating and the consequent evaporation of crystallization or intramolecular water.

Aiming at getting greater accuracy of the results using the oven, it was decided to keep the material in uninterrupted drying for 24 hours, resulting in more reliable results for the moisture content by the conventional method, ensuring the complete drying of the material. Thus, the values obtained by drying in the oven were considered the standard values for the new method.

Figure 1— Standard procedure steps: mass definition, oven use and temperature check

(Source: Authors' personal files)

Three parameters were used to evaluate the accuracy of the moisture results obtained with the use of the microwave, in comparison with the oven method, namely: Pearson's correlation coefficient (***r***); the index of agreement (***d***), suggested by Willmott (1981), and the confidence index (***c***), which corresponds to the product between ***r*** and ***d***, according to the Camargo and Sentelhas' proposal (Camargo and Sentelhas, 1997). Pearson's correlation coefficient was determined by equation 1, while the index of agreement was obtained using equation 2:

$$\boldsymbol{r} = \frac{\sum_{i=1}^{J'}(O_i - \bar{O})(E_i^* - \bar{E}_i^*)}{\sum_{i=1}^{J'}(O_i - \bar{O})^2(E_i^* - \bar{E}_i^*)^2} \quad (1)$$

$$\boldsymbol{d} = 1 - \frac{\sum_{i=1}^{J'}(O_i - E_i^*)^2}{\sum_{i=1}^{J'}\left(\left|E_i^* - \bar{O}\right| + |O_i - \bar{O}|\right)^2} \quad (2)$$

Where: **J'** — number of observations; **O** — moisture value calculated using the oven; **E*** moisture value obtained by the microwave; $\bar{O}$ — average moisture values obtained using the oven; $\bar{E}^*$ — average moisture values achieved using the microwave.

Furthermore, the mean absolute error (***MAE***) suggested by Legates and McCabe Jr. (1999) was calculated according to Equation 3; the mean percentage error (***MPE***) of Chong et al. (1982) was calculated by Equation 4; whereas the root mean squared error (***RMSE***) was obtained by Equation 5.

$$\boldsymbol{MAE} = \frac{\sum_{i=1}^{J} |O_i - E_i|}{J} \quad (3)$$

$$\boldsymbol{MPE} = \frac{\sum_{i=1}^{J} \frac{|O_i - E_i|}{O_i}}{J} \text{x } 100 \quad (4)$$

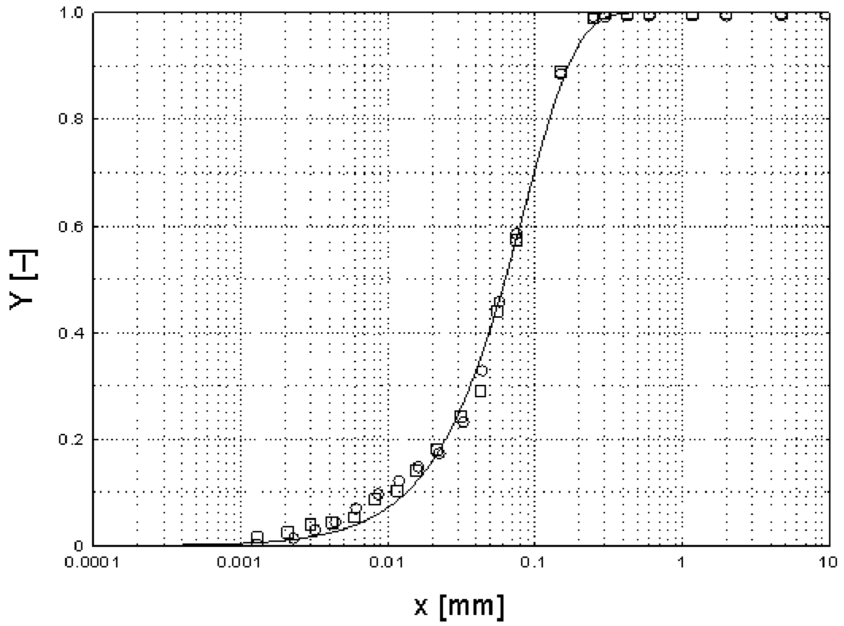
$$\boldsymbol{RMSE} = \sqrt{\frac{\sum_{i=1}^{J} (O_i - E_i)^2}{J}} \quad (5)$$

3 RESULTS

The determination of the sample density by picnometry showed the following values: 3,022.0 kg/m³ for the material from the temporary deposit and 3,068.0 kg/m³ for the material from the drain base. Such values are slightly high, but below the specific mass value of itabirite, of 3,710.0 kg/m³, a rock commonly mined in the region.

Analyzing the size distribution curves obtained from the tested material, and shown in Figure 2, the material is classified as sandy silt, being therefore equivalent to non-cohesive soil. Such soils have their moisture reduced faster during drying than the cohesive ones, allowing those common test conditions in terms of sample mass, initial moisture and number of samples during a test cycle.

Figure 2 — Particle size distribution of samples (circles: drain sample; squares: temporary deposit sample).



Regression analysis, using the computer package (freeware) SciDavis, as well as EasyPlot, the theoretical distribution that resulted with the highest statistical adherence was that by Rosin–Rammler–Sperling–Benett (or Rosin–Rammler, for brevity).

$$Y = 1 - e^{\left[ln\left(\frac{1}{2}\right)\times\left(\frac{x}{x_{50}}\right)^{m}\right]} \quad (6)$$

It can be seen that the two curves are practically equivalent. The regression values obtained are shown in Table 1.

Table 1 — Granulometric analysis with Rosin–Rammler regression

Provenance of bulk material	***Median* (*x_{50}*) [mm]**	***Sharpness* (*m*) [–]**	***Coefficient of determination* (*r^2*)**	***c^2***
Drain base	0.0628	1.2168	0.99808	0.00785
Temporary deposit	0.0634	1.2205	0.99803	0.00816

Castro et al. (2022) achieved good empirical correlation between the random packing porosity of spheroidal granular systems and the Rosin–Rammler sharpness coefficient. The theoretical porosity (spheroidal particles) is given by Equation 7.

$$\varepsilon(m) = 0.2204 \times \left[1 - e^{-\left(\frac{m}{1.1419}\right)^{1.4411}}\right] + 0.1503 \quad (7)$$

Applying the value ***m*** = 1.22 in preceding equation, the theoretical porosity (just in case of spheroidal particles) would be 29.7 %.

In turn, Lopes et al. (2020), following this line, have determined a theoretical prediction for the specific surface area (s_v) of a spheroidal granular system that can be described by the Rosin–Rammler equation (with sharpness coefficient equals to m). According to those researchers, the model can be systematized by the following three equations:

$$s_v = a(m) \times x_{50}{}^{-b(m)} \tag{8}$$

Where, the preexponential parameter can be expressed (with coefficient of determination: r^2 = 0.9991) by Equation 9.

$$a(m) = \frac{290.0568 \times m^{-1.65813}}{(m^{-1.65813} + 0.20708^{-1.65813})} + 6 \tag{9}$$

And the exponent of equation (8) can be calculated (with coefficient of determination r^2 = 0.9997) by Equation 10.

$$b(m) = 1 - exp\left(-\left(\frac{m}{0.67235}\right)^{1.46902}\right) \tag{10}$$

Using the median and mean sharpness coefficient of the systems under study, the value of 44.54 m²/kg is found for the specific surface area (referred to the mass of solids, as usual). Table 2 systematizes the results.

Table 2 — Prediction of the specific surface area of a spheroidal system with an equivalent particle size distribution.

Preexponential parameter: ***a(m)*** =	20.5545 m²/m³
Exponent: ***b(m)*** =	0.909241
Specific surface area (volumic): s_v ***(m, x₅₀)*** =	13,562.0 m²/m³
Specific surface area (mass): s_m ***(m, x₅₀)*** =	44.54 m²/kg

The effective use of a microwave oven for moisture content measurement depends on a number of interrelated factors, such as: (i) material type, (ii) running wattage, (iii) exposure time and temperature , (iv) sample size and use of multiple simultaneous samples, (v) containers used in the test, and (vi) possibility of alteration of material properties caused by exposure to microwaves (Usmen and Kheng, 1986).

Therefore, Jastrzębska, (2019) recommends that the drying time in a microwave oven be adjusted to the sample's mass, its moisture content and the number of samples disposed inside the device during a drying cycle. On the other hand, Mafuma and Muller, (2020) have pointed out that considering the use of a microwave, the drying time does not significantly depend on the sample size and its initial moisture content.

From the experimental data of moisture determined by conventional oven and microwave testing, calibration curves were studied by linear and non-linear regression (a power function). For the linear regression to predict the true value (of the type $y = a \times x + b$ here considered as the standard assay, based on the microwave assay, it results in angular coefficient a = 1.0296, and intercept b = -0.0025. The coefficient of determination was R^2 = 0.9953 which is very good. In turn, the regression using a potential function (type $y = c \times x^d$), using the staggered algorithm of Levenberg–Marquardt (using the Software SciDavis) with tolerance of 0.0001, resulted r^2 = 0.9973 but it did not prove to be advantageous (regression values were: c = 1.08353 and the exponent d = 1.03286).

Figure 3, which is the comparison between the averages of the moisture contents obtained with the microwave oven and the conventional oven, illustrates this well. The circles are the experimental values, the dashed line represents the linear regression, while the crosses refer to the values predicted by the potential equation. Figure 4 illustrates the average relative deviation of the estimates (relative to the values obtained in the electrical oven); it evidences, in a similar way, that there is no apparent gain in adopting the non-linear regression (power equation). It can be seen that in the region below 5.0 % (moisture range in which the geotechnical consequences are not critical) there is lower accuracy of the microwave-based method.

Figure 3 — Calibration of average moisture data obtained using microwaves versus values obtained using a conventional electrical oven

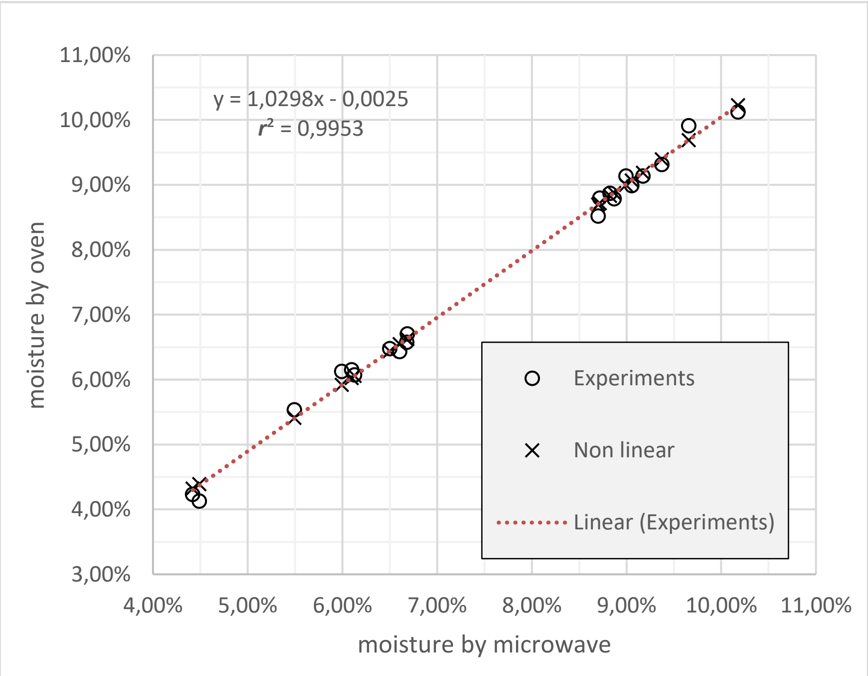


Figure 4 — Mean relative deviations of theoretical estimates from microwave in relation to the corresponding values obtained with the use of a conventional electrical oven (circles: power equation; squares: linear equation)

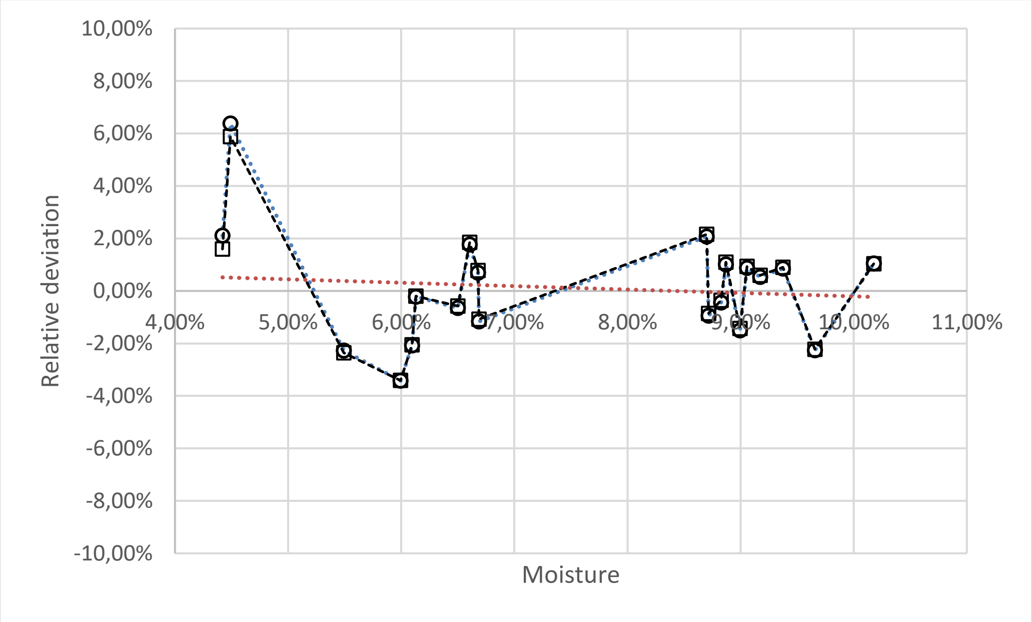


Table 3 summarizes the results of the microwave performance indicators, using linear regression. The Pearson correlation coefficient has value of 0.9976; mean absolute error of 0.1078; root mean square error of 0.1376 and mean percentage error of 1.69 %.

Table 3 — Statistical analysis of moisture data using an oven and microwave (linear regression)

Parameters	***Results***
Pearson's coefficient of correlation (***r***)	0.9976
Index of agreement (***d***)	0.9985
Confidence index (***c***)	0.9961
Mean absolute error (***MAE***)	0.1078
Mean percentage error (***MPE***)	1.69 %
Root mean squared error (***RMSE***)	0.1376

4 CONCLUSIONS

The suitability analysis of the use of the microwave oven to determine the moisture content aimed to validate a test method that allows the assessment of the geotechnical condition of every stacked layer of tailings on disposal areas more quickly.

As verified in this study, the use of the microwave oven following the ASTM D4643-17 standard for filtered iron ore tailings has showed linearity in the results when compared with the conventional oven method. However, the recommended method has shown less accuracy in the range of low moisture values (below 5%), which does not represent a critical disadvantage from the point of view of geotechnical safety (in terms of pore pressure or risk of liquefaction).

The gains in the operational cycle and in the technical quality of the results are of great importance for the development of technological control techniques in compacting of dewatered tailings. The economic impact of the method advocated here will be great, since the movement of large volumes of tailings in limited areas leads to customary downtime, often resulting from waiting for confirmatory analytical results to practice within the safety limits. Faster testing, therefore, leads to less non-productive time.

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