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Rice – Determination of the potential milling yield according to ISO 6646, evaluation of lipid content and colour Kett index.

Edizioni Nuova Cultura

Rice – Determination of the potential milling yield according to ISO 6646, evaluation of lipid content and colour Kett index

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Abstract: Milling of rice plays a key role in determining rice quality and value. Rough rice milling is an important process in which rough rice is milled to produce milled or polished grain to meet the consumer's preference. Actually, the standard method for the determination of the yield of husked rice is the ISO 6646:2011. In this study the laboratories of various Italian organizations of the rice sector were involved; they applied the ISO 6646:2011 standard on Italian paddy rice varieties: Caravaggio, CL26, Volano and Sole CL. The following were evaluated: the applicability of the ISO 6646:2011 standard, the consistency of the performance data with the needs of the Italian market and the correlation of the degree of milling with the lipid content and the colour index.

Key words: rice, potential milling yield, lipid content, Kett colour index

Introduction

Rice is used as wholegrains, hence any breakage of the grain during milling is undesirable. The primary reason for milling breakage lies in defects of the grain entering the mill, mainly in grain cracks. The rice grain is mechanically strong, but is susceptible to moisture stress and develops fissures upon rapid hydration or dehydration either in the field or in the process of drying. Gentle milling can prevent defective grains from breaking, while harsh milling leads to failure of more or all of them (Bhattacharya, 2011).

The process of rice milling is probably nearly as old as agriculture itself, but mechanized rice milling traces its roots back to the invention of an abrasive type of bran removal equipment, invented by Douglas and Grant around 1860. Since then, rice milling has evolved into a complicated industrial process that employs sophisticated technologies for controlling the whole process (Champagne, 2004).

Rice milling is a process in which people use machines to remove foreign material, husks, bran, and broken kernels from rice to prepare the grain for a variety of commercial purposes (Champagne, 2004).

Most rice is milled for direct consumption or for subsequent utilization as an ingredient in end-use products. The primary purpose of milling is to remove the germ and bran layers from the kernel endosperm. The extent to which bran has been removed from the kernel is referred to as the "degree of milling" (DOM). Generally, the milled-rice customer and the intended use of the rice dictate the target bran removal level (e.g., most rice milled for breakfast cereal processing is not milled to the same extent as that used for "table" rice) (Champagne, 2004).

The four morphological layers surrounding the rice kernel endosperm (the pericarp, seedcoat, nucellus, and aleurone layers) and the germ are hereafter collectively referred to as "bran". The bran contains approximately 18-20% lipids and 14-15% protein, while milled rice, comprising primarily the kernel endosperm, is generally much lower in lipids (approximately 0.3-0.5%) and protein (approximately 7%). These values can vary greatly due to varietal, environmental, or processing variability. Because of these stark differences in composition between the bran and endosperm, the DOM can affect the functionality of milled rice. In addition to having functional effects, the bran remaining on kernels after milling can affect sensory characteristics (Champagne, 2004).

Milling quality is composed of several factors that directly affect the value of rough rice. It encompasses the total amount of milled rice recovered after milling (total milled-rice yield) and the total amount of whole kernels recovered after milling (head-rice yield or whole-rice yield). The purity of the rough-rice samples is also a component of milling quality. Specifically, the value of rough rice samples is negatively affected by the presence of cracked kernels, red rice, discolored kernels, or immature kernels (Champagne, 2004).

Abrasive testing mills are used to determine milling quality. They are important tools used by rice breeders to assess the milling quality of breeding progeny, by scientist to prepare samples for study, by government agencies to determine the grade of rice, and by processors to set the price of rice. Differences between rice laboratory mills mainly rely on the principle behind the milling process, i.e., friction versus abrasion, the amount of sample required and whether dehulling is first needed (Champagne, 2004).

Potential milling yield

Determination of the potential milling yield

Brown rice milling yield is determined by expressing dehulled rice as a weight percentage of rough rice. This measurement also indicates the amount of hull in a given sample. Total milled-rice yield is determined by expressing combined broken and whole-kernel yield as a weight percentage of rough rice. Head-rice yield is the yield of milled rice that is three quarters or more of normal kernel length, expressed as a percentage of rough rice or total milled rice. The most common method for determining whole vs. broken kernels is to place the sample on a shaker table, which consist of two inclined indent plates that vibrate. Kernel weakness that results in breakage during milling is reported to be related to fissuring (cracking), chalkiness, and kernel dimensions (Champagne, 2004).

An important aspect of milling quality is the degree to which a given sample has been milled. Degree of milling is a quantification of the amount of bran removed from kernels during the milling process. The majority of consumers around the world prefer well-milled rice that has a little to no bran remaining on the kernels (Champagne, 2004).

Standard reference

Measurement of milling quality in the laboratory requires a high-quality laboratory mill, which can provide consistent milling performance for a given rice sample, enabling the comparison of milling quality among different varieties. In addition, the milling quality measured with a laboratory mill can simulate the milling performance of a large rice lot in industrial-scale system, enabling new rice varieties to be easily accepted by the commercial milling industries (Bao, 2019).

Actually, the standard method for the determination of the yield of husked rice is the ISO 6646:2011.

This International Standard specifies a laboratory method for the determination of the yield of husked rice obtained from paddy or parboiled paddy (*Oryza sativa* L.), and for the determination of the yield of milled head rice obtained from paddy or parboiled paddy, or from husked rice or husked parboiled rice. This International Standard is only applicable to abrasive milling equipment (ISO 6646:2011).

The ISO 6646 provides that the husk is mechanically removed from paddy. The resultant husked rice is then weighed. Next, the bran and germ are mechanically removed from the husked rice to a fixed reduction in mass and the resulting milled head rice is weighed (ISO 6646:2011).

Milling quality

Is very important to consider that unmilled (brown), rice compared to milled rice, contains more protein, lipids, vitamins, minerals, and phytochemicals with potential health benefits (Champagne, 2004). It follows that the content of the lipids is an indirect measure of the degree of milling processing (Simonelli et al., 2013).

Lipid content

Lipids, although not as abundant as the carbohydrate and protein components, are important in rice because they contribute to nutritional, sensory, and functional qualities (Champagne, 2004).

Lipids are present in the form of spherosomes, or lipids droplets, with diameters of <1.5 mm in the aleurone layer, <1 mm in the subaleurone layer, and < 0.7 mm in the embryo of rice grain. Most of the lipids in the endosperm are associated with protein bodies, but it is believed that starch granules also have bound lipids. Lipids are generally classified into nonstarch lipids, principally those in the spherosomes of the aleurone layer and embryo, and starch lipids (Champagne, 2004).

During the milling process nonstarch lipids, or superficial lipids, are principally removed.

Colour index

Consumers prefer kernels that are very white rather than inherently gray or discolored from environmental effects such as stink bug infestation or kernel smut. Whiteness can be measured using different meters (such as Kett analyzer) as well as digital image-analysis systems. In specific literature (i.e. *Rice Chemistry and Techology*) is reported that "inherent kernel whiteness should not be confused with degree of milling". That is, differences in degree of milling affect kernel whiteness, but when various samples are milled to the same degree of milling they can still show variation in whiteness (Champagne, 2004). For example, a study was conducted on a typical rice risotto variety grown in seven distinct different Italian areas. All the seven samples were milled in the same way and at the same DOM: nevertheless, the intensity of color of the grains measured by the Kett analyzer were different, being dependent to territoriality (Galassi et al., 2013).

The aim of the work

The purpose of this study was to evaluate the applicability and reliability of the ISO 6646 standard in various Italian organizations that routinely perform the yield test, but usually with internal methodologies and not applying the ISO standard. We wanted to understand if the different operators obtain results that are comparable with the laboratory mills usually used (the standard is in fact generic and does not specify which type of laboratory mills should be used), applying ISO 6646:2011.

MATERIALS AND METHODS

Laboratories

Seven laboratories took part in the study (Ente Nazionale Risi - ENR, Camera di Commercio di Biella e Vercelli, Agenzie delle Dogane di Savona, Riso Scotti, Curtiriso, Riso Gallo, CREA-CI); they usually perform milling process on rice, but never applying the ISO 6646:2011.

Materials

Four husked Italian rice samples (Volano, CL26, Caravaggio, Sole CL) were considered, whose characteristics and classification, according to EC Reg. 1308, are reported in Table 1.

Characterization

Milling process

All the participating laboratories followed the procedures described in ISO 6646:2011, after checking the adequacy of their available equipment (Universale – L1, L2, L3, L4, L6, L7 - and Satake – L5 – abrasive testing mill).

According to ISO 6646, paddy (a minimum of 200 g) was dehusked in the testing husker. The total yield of husked rice was weighed to the nearest 0,01 g.

The husked rice was divided to give a portion suitable for the equipment; the mass was weighed and recorded the nearest 0,01 g (a minimum of 100 g is recommended). The husked rice sample was introduced in the testing mill and milled for the time necessary to remove the mass fraction (10 ± 0,5) % of its total mass. The milled rice obtained was weighed and the mass recorded to the nearest 0,01 g. Head rice was separated from the broken kernels, placed in separate bowls, and weighed, its mass being recorded to the nearest 0,01 g. The test was carried out in duplicate.

Lipid content - Determination of crude fat

For crude fat, the Soxhlet extraction method with petroleum ether as solvent was used, according to AACC Method 30-25.01. 5 g of dried (in a vacuum oven: 100 mm Hg; 5 hours) grinded rice were extracted with 100 ml of petroleum ether (30-60°C) for 2 hours. The laboratory usually provides the lipid content analytical result by associating the analytical value with the estimated uncertainty with the metrological approach.

Kett – Colour index

The samples were analyzed by ENR through the application of an internal method (MP28 rev.01 2011) which provides for the triple reading of the whiteness of the whole milled rice grains inside the digital colorimeter instrument Kett C-300.

Table 1 – Characterization of rice

Variety	Classification	Other
Volano	Long A	Similar to Arborio
CL26	Long B	
Caravaggio	Long A	Similar to Carnaroli
Sole CL	Round	

Moisture content
Moisture content of rice samples was determined by using the standard methods of analysis ISO 712:2009. 5 g of grinded rice are dried in a Memmert UFE 400 oven for 2 hours at 130-133°C.

An alternative method for determining humidity is to use a thermobalance. The results in this case may be less accurate, but still indicative.

Table 4 shows the analytical choice of laboratories.

RESULTS AND DISCUSSION

Evaluation of performance of participants

Participants' performance was assessed using a numeric indicator, z-score, which is calculated as follows:

$$z = \frac{x - X}{\hat{\sigma}}$$

Where x is the result reported by a participant, X is the assigned value, which is the reference value obtained from the comparison, and $\hat{\sigma}$ is the standard deviation for proficiency assessment.

For each laboratory (and for each variety) the z-score was calculated and PT performance was assigned as follows:

$\lvert z\rvert \leq 2.0$	Satisfactory
$2.0 < \lvert z\rvert \leq 3.0$	Questionable
$\lvert z\rvert > 3.0$	Unsatisfactory

The X and $\hat{\sigma}$ values obtained are reported in Table 2 and the z-score is displayed in Figure 1.

By evaluating the global z-score graph (Fig. 1) it is evident that no unsatisfactory data (i.e. with a z-score value $\lvert z\rvert > 3$) are present. Only one data was questionable (z value -score: 2.09), but was not discarded in subsequent processing; hence, all data were accepted in the subsequent treatment.

The uncertainty of the assigned value, u_x, was estimated as reported in the ISO 13528:2015 standard, as follows:

$$u_x = 1.25 \cdot \frac{\sigma_p}{\sqrt{p}}$$

where: σp represents the standard repeatability deviation of the PT, or the robust standard deviation, shown in Table 2.

p represents the number of data processed (7).

Performances of the method

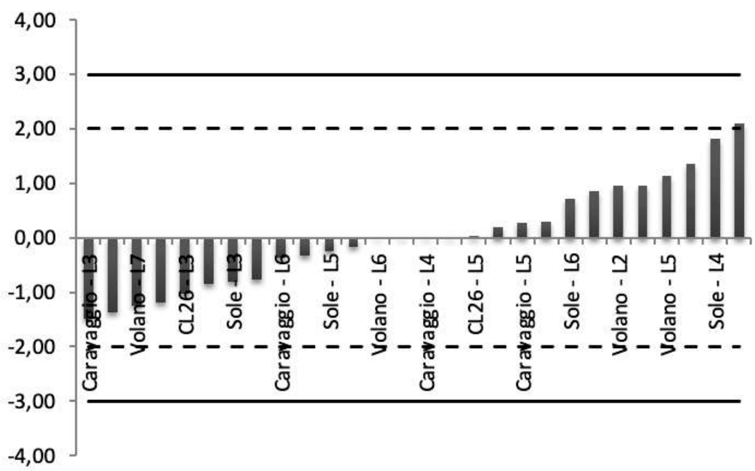
Values of f
The prescription provided to all the laboratories participating in the PT was to maintain a value of removed mass fracion $f = 10.0 \pm 0.5$ (see 8.1.2, ISO 6646:2011). All operators maintained the values of f between a minimum of 9.5 and a maximum of 10.5, except for L2, which has always obtained slightly lower f values, and L6, which deviated from the defined range.

Moisture of the samples
Before performing the milling yield, ISO 6646 ex-

Table 2 – Characterization data of the proficiency assessment

Variety	Data number p	Assigned value X	Uncertainty u_x	Standard deviation σp
CL 26	7	79,1	0,7	1,6
Volano	7	81,4	0,9	1,9
Caravaggio	7	81,3	0,5	1,0
Sole CL	7	85,2	0,7	1,4

Figura 1 – z-scores values



pressely requires the determination of the moisture content of the samples, that should be 13.0 ± 1.0 g / 100g d.m. In our case, it also becomes a parameter for verifying the homogeneity of the distributed samples. In our interlaboratory trial, moisture content deviated from what is defined by the standard (Tab. 4) for Volano, CL26 and Caravaggio, but quite in the ranges established by the standard. For the SoleCL variety, on the other hand, the humidity range of the samples established is respected.

Evaluation of the method's precision data (ISO 6646: 2011)

As it was not possible to identify trends for the repeatability and reproducibility reported in the ISO 6646, the following criteria, reported in ISO 6646:2011, were assessed:

- repeatability: for huscked rice: 1%; for milled head rice: 2%
- reproducibility: for huscked rice: 3%; for milled head rice: 5%

Verification of the validity of the results

Repeatability

Not all participants provided information on the yield in husked rice. It was not possible for everyone to carry out this data processing, conducted only by the laboratories L3 and L5. The results are shown in the left part of Figure 2. The same evaluation was carried out for milled head rice and is reported in the right part of Figure 2. It is possible to notice that for all the laboratories and for all the rice varieties, the repeatability criteria defined in the standard were respected.

Table 3 – mass fraction removed, f

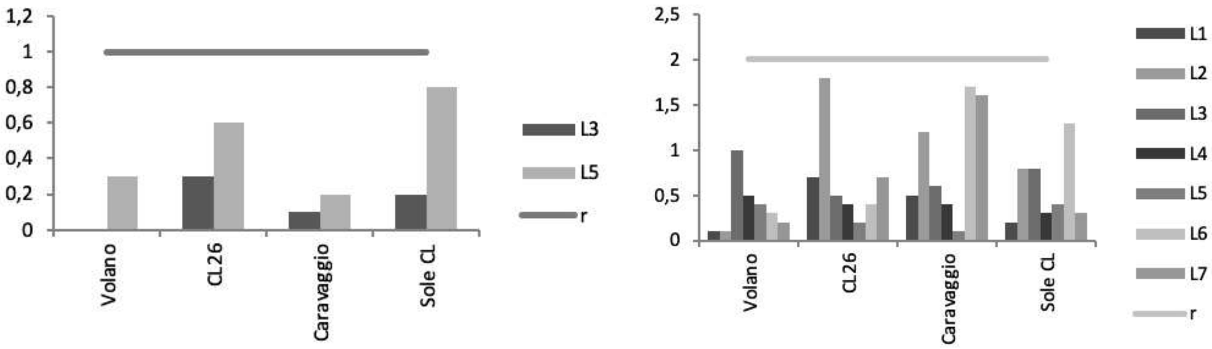
Laboratory	Volano	CL26	Caravaggio	Sole CL
L1	9.7	10.0	10.0	9.5
L2	9.4	8.7	9.0	8.9
L3	10.0	10.3	10.2	9.9
L4	10.2	10.2	10.1	10.2
L5	10.1	10.1	10.1	10.1
L6	10.6	9.0	11.1	8.8
L7	10.3	9.6	9.7	10.0

Table 4 – moisture content (% d.m.) of brown rice

Laboratory	Volano	CL26	Caravaggio	Sole CL
L1	12.59	12.13	13.29	13.55 [2]
L2	12.29	11.94	12.63	12.58 [1]
L3	12.09	11.74	12.67	12.46 [1]
L4	12.29	11.94	12.63	12.58 [1]
L5	12.38	12.20	12.99	12.97 [2]
L6	11.80	10.70	11.90	12.50 [2]
L7	12.50	11.00	13.60	12.50 [2]
range	*11.80 – 12.59*	*10.70 – 12.13*	*11.90 – 13.60*	*12.46 – 13.55*
	0.79	1.50	1. 70	1.09

1. in these laboratories the moisture was carried out according to the official ISO 712 method
2. in these laboratories for the determination of moisture, a rapid method using a thermobalance was used

Figure 2 – repeatability for husked (left) and milled head rice (right)



Reproducibility

In order to compare the data of the different participants (Table 5), the minimum and maximum yield value for each variety was assessed and the acceptability of the requirement compared with the reproducibility limit equal to 5%. It is possible to note that for Volano, Caravaggio and Sole CL the criterion is always respected. while CL26 was out of the reproducibility range. This non-acceptability can be due to several factors. The sample humidity is too low and out of the range defined by the standard; maybe the heterogeneity of the processing factors *f* influenced the results ,or the variety is particularly heterogeneous. The R = 5% value is in fact valid only for samples with a moisture content in the range 13.0 ± 1.0 g / 100g and with *f* = 10.0 ± 0.5 (see Tables 3 and 4). It is not possible to assert that the criterion is not satisfied due to incorrect operation or the standard itself, as for the other three samples the criterion is largely satisfied, in particular for Volano and Caravaggio.

Evaluation of other analytical parameters

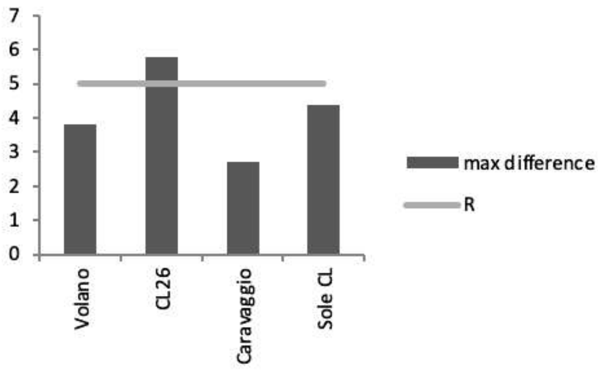
Degree of white (Kett)

It is known that the degree of milling of rice is closely connected with the lipid content (Simonelli et al., 2013), but also with the coloring of the rice grain, which can be precisely measured, through the Kett

Table 5 – Milled head rice value

	Volano	CL26	Caravaggio	Sole CL
L1	77.8	81.4	80.0	85.2
L2	80.6	81.1	82.6	85.5
L3	77.7	79.5	79.9	84.1
L4	80.6	83.0	81.3	87.9
L5	80.9	81.5	81.6	84.9
L6	79.1	85.3	81.0	86.3
L7	77.1	80.8	81.6	83.5
min	77.1	79.5	79.9	83.5
max	80.9	85.3	82.6	87.9
diff	3.8	5.8	2.7	4.4

Figure 3 – reproducibility for milled head rice



colorimeter as white index (gdb Kett). Through the evaluation of analytical historical data, it is possible to assert that a commercial sample of milled rice has a gdb Kett approximately in a range between 45 and 50. The gdb Kett was determined for all the samples derived from the milling yield trial and the results are reported in Table 6.

It is possible to note that for the Volano variety, there are 7.9 points of range of gdb Kett between the different samples. The L2 laboratory was the one that found a gdb Kett value significantly lower than the others (darker sample); in fact it was the laboratory that removed less mass fraction than the others (f = 9.4).

As for the CL26 variety, gdb Kett ranged by 7.2. It is possible to note that the samples corresponding to L2 and L6 are those that have the lowest gdb Kett values compared to the others and are therefore slightly darker; in fact they undergone a lower processing (respectively f = 8.7 and f = 9.0), lower than the range established by the ISO 6646 standard for the value of f.

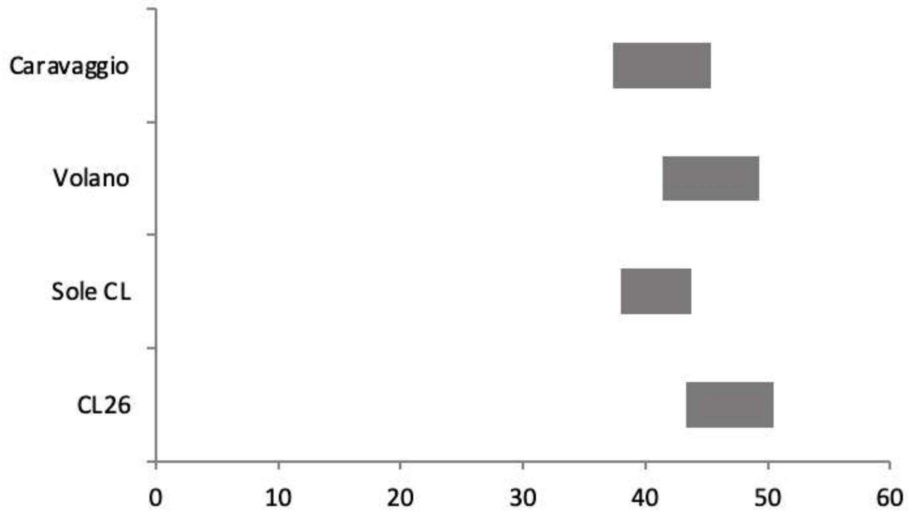
Evaluating the Caravaggio variety, the range of the gdb Kett between the different samples is 8.0. Again the sample corresponding to L2 provided the lowest gdb Kett (37.3) and was, consistently, also the one with less mass fraction removed (f = 9.0). The sample with f = 11.1 (the higher of the series) is L6 which, in fact, has the highest gdb Kett value (45.3) among all the Caravaggio variety.

Finally, as regards the Sole CL variety, L2 and L6 showed the lowest values of the gdb Kett, consistently with their f values of 8.9 and 8.8 respectively. For this variety the range of variation among laboratories was the smallest (5.7).

Table 6 – gdb Kett on rice samples milled according to ISO 6646 (with f = 10).
The values reported are the average of the two sub-samples

	Volano		CL26		Caravaggio		Sole CL	
	average	IF	average	IF	average	IF	average	IF
L1	46,3	0,9	39,8	0,4	43,6	0,7	41,8	0,7
L2	41,4	1,5	34,3	0,4	37,3	1,3	38,0	0,7
L3	46,4	0,2	41,1	0,7	43,5	0,6	40,6	0,6
L4	46,9	1,0	41,6	0,9	41,9	1,1	42,2	1,4
L5	46,5	0,8	41,4	0,3	44,4	0,5	43,6	0,2
L6	46,0	1,3	37,2	0,3	45,3	0,2	40,1	0,7
L7	49,3	0,5	41,1	0,4	44,0	0,8	43,2	0,3
range	*41.4 - 49.3*		*34.3 – 41.6*		*37.3 – 45.3*		*38.0 – 43.6*	

Figure 4 – range of the gdb Kett on different rice varieties processed according to ISO 6646 (f = 10)



In Figure 4 it is possible to see how different varieties processed in accordance with ISO 6646, with a similar processing degree (f = 10), provide levels of gdb Kett (intended as range) that can differ significantly from each other. The rice variety Volano, for example, turns out to be significantly darker than the CL26. This may be due to the varietal characteristic itself, but also to environmental factors linked to the growing place.

Lipids content

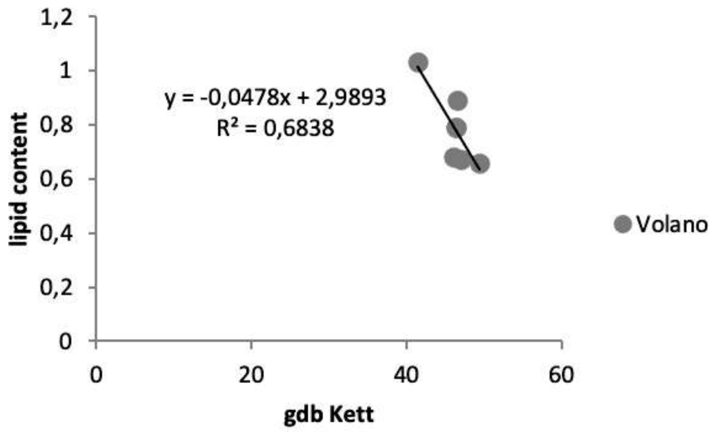
The lipid content of the Italian rice varieties analyzed is reported in Table 7. For each variety it is possible to correlate the lipid content as a function of the gdb Kett (Figure 5) and then the gdb Kett or the lipid content as a function of *f* value.

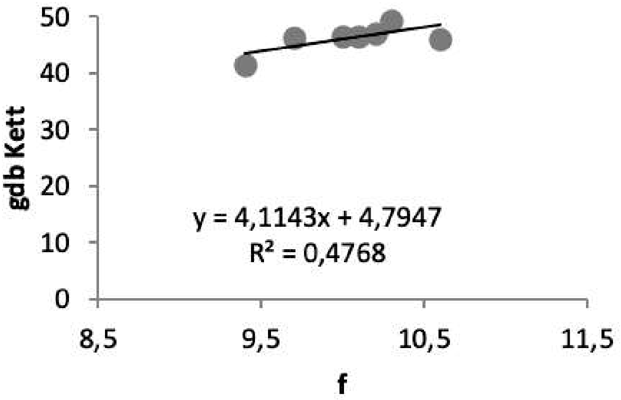
It is possible to note the inverse correlation between the lipid content and the Kett gdb. This is expected since the higher the milling degree of

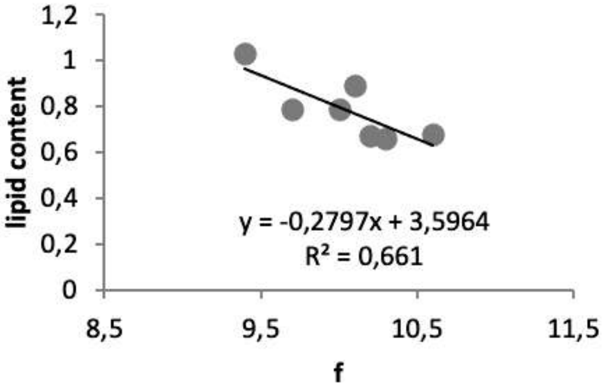
Table 7 – lipid content on rice samples milled according to ISO 6646 (with f = 10)

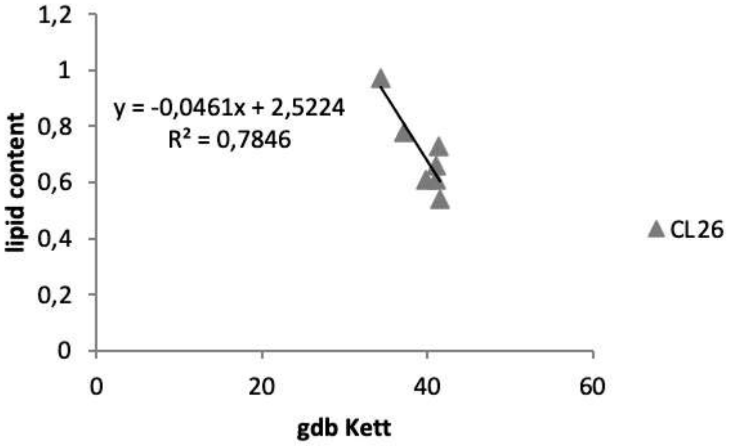
	Volano	CL26	Caravaggio	Sole CL
L1	0,79	0,61	0,77	1,02
L2	1,03	0,97	0,92	1,01
L3	0,79	0,66	0,90	1,07
L4	0,67	0,54	0,83	0,93
L5	0,89	0,73	0,89	1,15
L6	0,68	0,78	0,61	0,95
L7	0,66	0,61	0,93	0,92
range	*0.66 – 1.03*	*0.54 - 0.97*	*0.61 – 0.93*	*0.92 – 1.15*

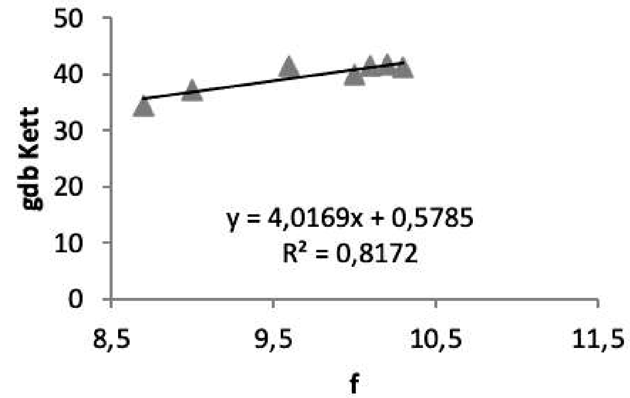
The uncertainty, estimated by the Laboratory using the metrological approach, for all samples is equal to 0.06%

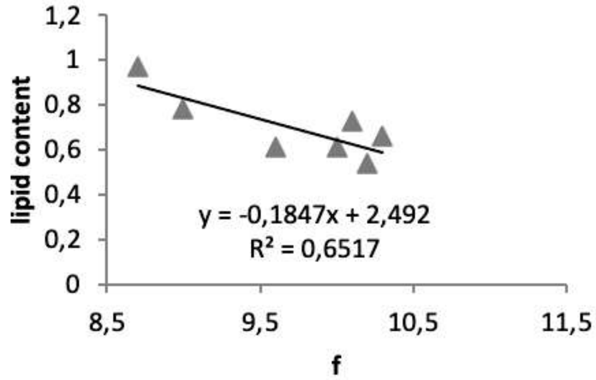

1,2
1
0,8
0,6
0,4
0,2
0
lipid content
y = -0,0478x + 2,9893
R² = 0,6838
Volano
0
20
40
60
gdb Kett

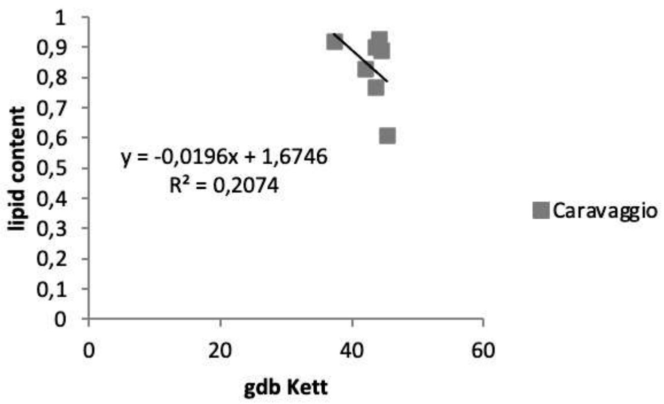

50
40
30
20
10
0
gdb Kett
y = 4,1143x + 4,7947
R² = 0,4768
8,5
9,5
10,5
11,5
f

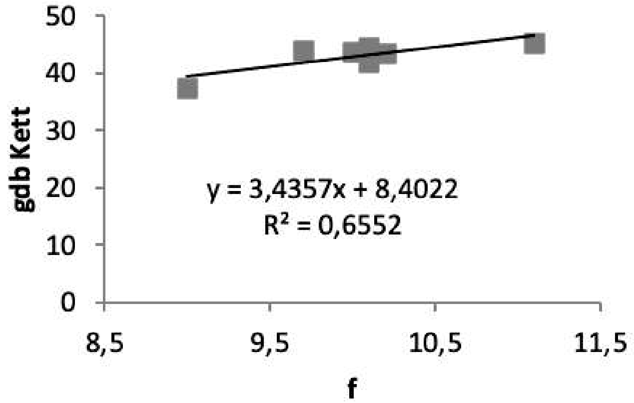

1,2
1
0,8
0,6
0,4
0,2
0
lipid content
y = -0,2797x + 3,5964
R² = 0,661
8,5
9,5
10,5
11,5
f

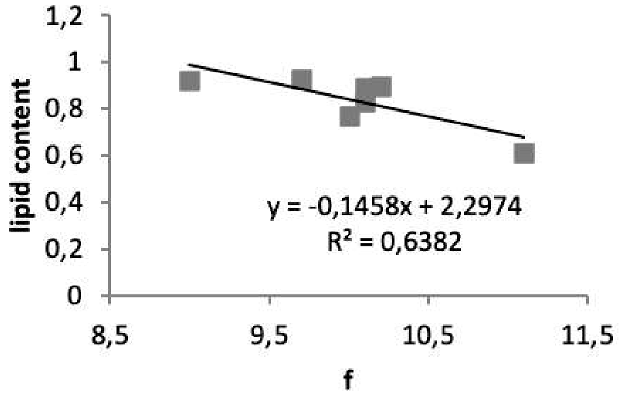

1,2
1
0,8
0,6
0,4
0,2
0
lipid content
y = -0,0461x + 2,5224
R² = 0,7846
CL26
0
20
40
60
gdb Kett


gdb Kett
y = 4,0169x + 0,5785
R² = 0,8172
f


lipid content
y = -0,1847x + 2,492
R² = 0,6517
f


lipid content
y = -0,0196x + 1,6746
R² = 0,2074
Caravaggio
gdb Kett


gdb Kett
y = 3,4357x + 8,4022
R² = 0,6552
f


lipid content
y = -0,1458x + 2,2974
R² = 0,6382
f

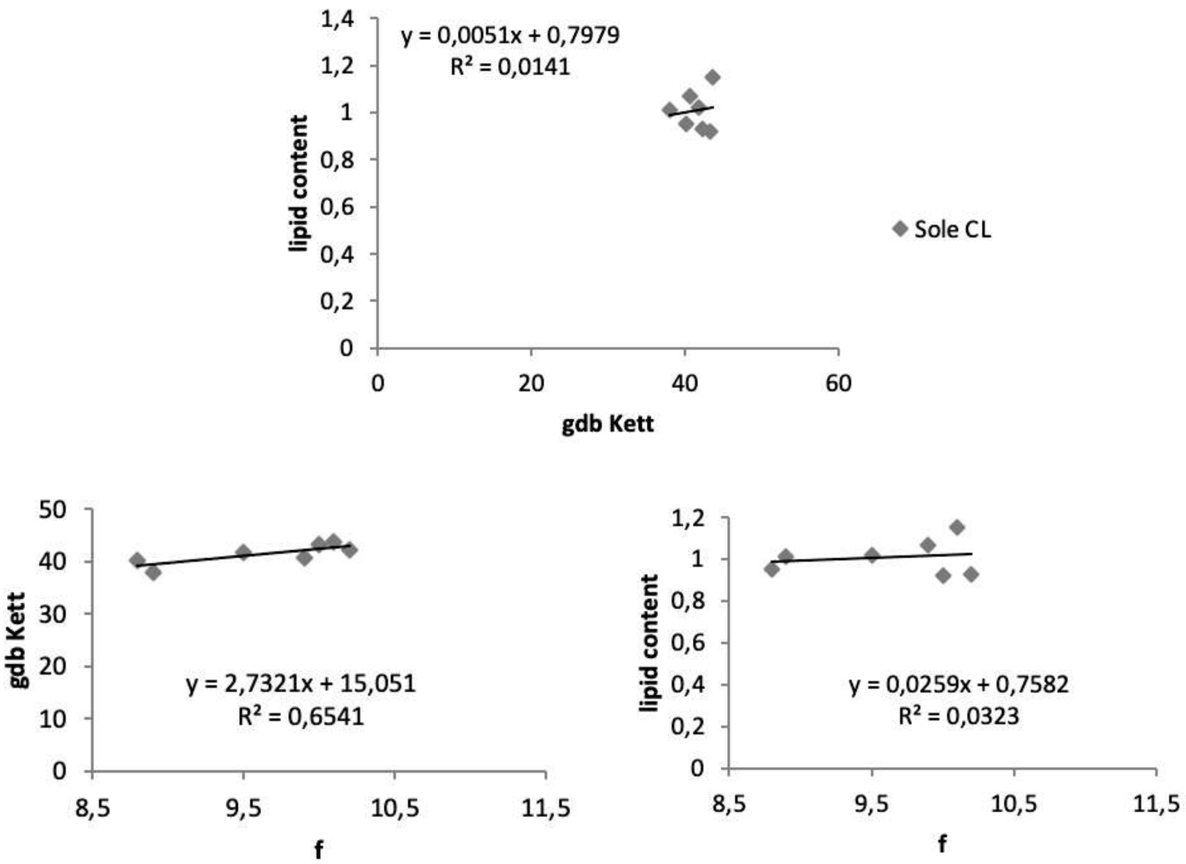


Figure 5 – graphical correlation between lipid content, gdb Kett and f value

rice, the clearer it is. The gdb Kett is directly proportional to f, even if the one taken into consideration has a very small range. Obviously, on the other hand, the lipid content appears to decrease as the milling degree increases (the lipid content is inversely proportional to f). Among the various varieties considered, the Sole CL responds less sensitively than the others to the different degree of processing, understood as the lipid content; his excursion is in fact the smallest.

Conclusions

Based on the performance assessments of the ISO 6647: 2011 standard, we demonstrated that it is applicable by all operators, with good repeatability results that fall within the limits defined in the standard. As for the reproducibility of the method, it has given satisfactory results, since the comparison of the data of all the operators remained within the reproducibility limit of the standard, although different testing mill have been used, with different operators, in different times, sometimes with different *f* and the humidity was slightly outside the value of 13.0 ± 1.0 g / 100g. Only for CL26 sample the reproducibility data was not respected (5.8 was found on a limit of 5). We suppose that, if subjected to conditioning to get humidity in the range 13.0 ± 1.0 g / 100g, its milling performance could have been better.

Despite these encouraging results, ISO 6646: 2011 turns out to be too complex and laborious for its routine use, therefore it is not applied in

Italy, where a method of simpler application and with better performance results is desirable.

It was confirmed that the Kett gdb is a parameter of simple determination and usability, which provides results related to the degree of milling. Although it can't be used alone in absolute terms, it can be useful for inferring milling yield. As expected, we showed that the lipid content can very well describe the degree of milling.

It would be interesting to continue the study undertaken here with other values of *f* also to evaluate the correlation with lipids and gdb kett.

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