



Evaluation, ranking and positioning of measurement methods for pellet production

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ABSTRACT

Pellet production and consumption are steadily increasing as a renewable energy source. The production and combustion properties of pellets are defined by molecular structure and elemental composition of raw materials. Quality control tools are different in terms of areas they cover the pellet-production cycle, but considering the raw materials, they regulate only the origin but not the components. There are standardized methods for measuring the biomass and these methods are mainly capable to the pellet raw material qualification, too. Using these together with the control and diagnostics of production parameters, the finished pellet quality (parameters) can be forecasted with high accuracy. A novel evaluation methodology is proposed in the paper for the measurement and qualification of the raw material. The introduced evaluation ranks these methods, based on measuring device-needed, time-requirement and measurement complexity triad. Moreover, the proposed best measurement solutions are positioned along the pellet production chain.

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1. Introduction

Pellets are special kinds of biomass-based biofuel. The speciality of this product is, that they have high energy density, low moisture content and uniform shape at the same time [1][2]. These features can provide nearly the same comfort level of application, as natural gas-based heating system [3].

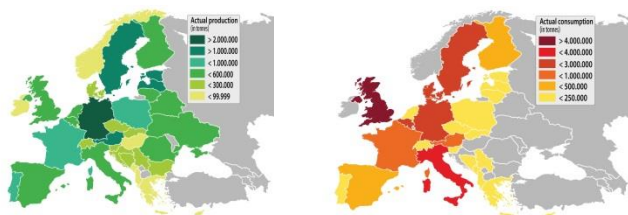


Fig. 1. European wood pellet production (left) and consumption (right) in 2015. [5]

Favourable characteristics of pellets are contributing to the continuously spreading of their consumption. It is evident by the fact, that over the past 15 years, nearly thirty-fold increase is measured considering the amount of consumed pellets in Europe [4][5].

In 2015, 50% of world production of wood pellets (14.1 million tons) happened in the EU, and in the same time 70% (20.3 million tons) was consumed here [6]. Distribution of the European pellet production and consumption in 2015 is presented in Fig. 1. Both the production and the consumption of pellet show a continuously growing trend. The annual amount of produced pellet in the EU has increased by 4.7% and the amount of consumed pellets by 7.8% from 2014 to 2015. [6][7]. The amount of consumed pellet is small percentage (0.6%) of the EU's primary energy consumption [8], but the pellets are valuable and evolving energy sources, which fits in the energy policy of the European Union according to security of supply, competitiveness, and sustainability aspects [9]. Since pellets are relative young energy sources, lots of questions arise about pellet production and

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consumption today and answering them requires further intensive research activities.

2. Pellet production

During the pellet production, with use many types of biomass raw materials, a compact, cylindrical shape, low moisture constant and high calorific value biofuel is produced [10].

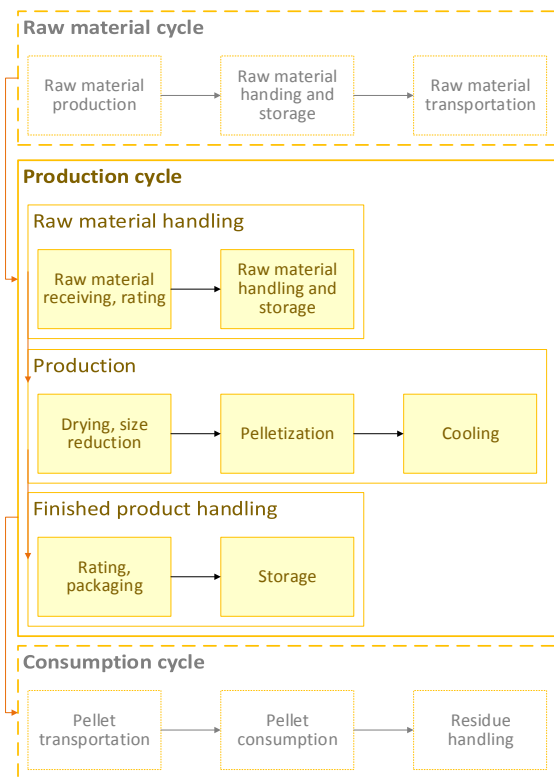


Fig. 2. Pellet production cycle

The whole pellet production cycle is complex. It incorporates a variety of raw material production, their handling and transportation; the pre-produced raw material treatment and the finished product manufacturing. Packaging has predefined quality classes and storage; the transfer of the finished product to the end-user, and finally, the residues handling, too, as represented in Fig. 2. [10].

2.1. Production cycle

After the arrival of the raw materials they have to be stored and handled. Studies confirm that the storage time of raw materials has effects to the finished product quality parameters [12]. Microbiological and chemical processes are the root causes of this effect - which are dependent of the raw material molecular structure, elemental composition and moisture content, as well as of the storage mode and conditions [13][14]. These processes may result in negative effect to the raw material quality, consequently, to the finished product, too. The raw material drying and grinding processes influence its moisture content, too. The

optimum moisture value is defined by parameters of the raw material mixture, and it is 10-14% usually [3][15]. The pellet production requires 2-4 mm sized, fine materials, and optimum pressure has to be applied in order to reach appropriate compressibility and evolving natural lignin-based bindings. During pressing the material through the die, the temperature is increasing due to the friction and without using auxiliary materials, natural material bonding can be realized. As the main factor, the moisture content has an effect on coefficient of friction. Also it has effects on the generated heat and the finished material bonds quality, too. The temperature of the finished pellets is high, and pellets are in a fragile state in this condition. Pelletability and combustion properties are decisively influenced by the raw material parameters and the production processes, too [3][18][19][20].

These quality factors are critical parameters. Certified biofuels satisfying the current standards can be manufactured only with optimization and precise control of the raw materials on one side and the production processes, on the other.

3. Pellets' standardization and final product quality parameters

Pellets are compressed organic fuels, which are typically prepared from wood as raw material. New raw materials had been involved in production, in the order to satisfy the greatly growing consumer demand [21][22]. These new, non-woody raw materials can be the following: herbaceous biomass, fruit biomass and aquatic biomass (e.g. algae). The quality of the raw materials is a crucial factor concerning the quality of the finished product, and the production processes, too [23]. In spite of the wood raw materials mixture, the non-woody materials compound has higher variation, which results great challenge to the pellet production industry [24] [3].

The International Organisation for Standardisation (ISO) has published the ISO 17225 (Solid biofuels - Fuel specifications and classes) standard series in May 2014. This series has replaced EN 14961 in November 2014. The EN ISO 17225 has wider scope than the previous standards, and is more useful according to the new non-woody raw materials, which has a greatly growing spread. The first part of the standard (EN ISO 17225 - Part 1) contains the general requirements related to biofuels. The second part of the standard (EN ISO 17225 - Part 2) includes property classes for wood pellets and the final part the same for non-woody pellets. The ISO standard regulates only the origin and source of raw materials; furthermore, it gives only categories based on possible application types (industrial or non-industrial application). However, the used raw material's quality is also determined by their molecular structure and chemical properties. So, the pelletability and combustion properties are influenced by the raw material features, too. Knowledge on these parameters is crucial for the regulation of the entire production process, furthermore, it may define also the quality of consumption [27][28].

Based on measurement results about the finished product's parameters, it is classified to additional quality classes (ENplus A1, ENplus A2 and ENplus B). The following list describes the final product's quality parameters:

- diameter and length,
- moisture content,
- ash content,
- mechanical durability,
- amount of fines,
- bulk density,
- net calorific value,
- amount of specified elements (Cl, N, S, As, Cd, Cr, Cu, Pb, Hg, Ni, Zn).

Table 1.
Scope of the quality control tools Standard

Standard	Raw material		Product		Purchase		Consumption
	Quality	Origin	Production	Quality	Transportation	Storage	
ENplus 3.0:2015*		✓	✓	✓	✓	✓	
ISO 17225:2014		✓	✓	✓			
EN 14961:2010		✓	✓	✓			
National standards*				✓			

* only for wood pellet

Quality control tools are different in terms of which pellet production cycle areas are covered [25][26]. The scope of the different quality tools is summarised in Table 1. The regulator and classifier tools do not cover the raw material quality and classes, moreover some standards neither regulates the activities between production and transfer to the end-user, nor the consumption. In the most comprehensive way ENplus standards covers the different areas of the whole product cycle. This standard was published by the European Biomass Association (AEBIOMA), but it doesn't contain regulation in relation to the raw materials, and its scope is just for wood pellets. *Resolving the regulation deficit on the raw materials is the main aim of the paper with introducing a novel methodology for finding the most appropriate measurement technique for the effective and efficient raw material control, together with the positioning of the best measurement methods along the pellet material flow.*

4. Measurement of biomass quality parameters

During the pellet production process the raw material is manufactured under relative high pressure and under the resulting higher heat. In thermogravimetric analysis [36], it was investigated whether these effects are causing a change

in the chemical composition of the raw material and the pellet produced therefrom. Since the temperature of the machined material typically does not exceed the degradation temperature of its main components, there is no significant difference between the raw material and the chemical composition of the finished product, which means that the composition of the finished product can be predicted with great precision [19][31].

There are various, standardized analytical methods for the general biomass qualification, which can be also suitable for the raw material qualification of pellets [29][30]. Using these methods, in addition to controlled production parameters, the finished product quality (parameters) can be forecasted with high accuracy, e.g. ordering the final product into the predefined classes of the ENplus standard [31]. These methods are featured usually by high device- and time requirement, as well as a high degree of complexity. The measurement samples preparation times are usually high, and there are only few methods, which can be fully automated.

The qualification and measurement of pellets' raw materials is receiving increasing importance and attention with the involvement of new biomass materials beyond wood and also with the application of "energy-woods", grown especially for energy usage, e.g. as raw materials for pellets, consequently, the efficient measurement of raw materials is becoming crucial. However, no recommendation or prescription is given in national and international standards or in scientific papers for selecting the most appropriate measuring method for pellets' raw material qualification. Such a proposal is one of the novelties introduced in the paper.

4.1. Key raw material parameters of pellets

At first the goal is to determine the raw material parameters that describe all of pellet main features, which may have significant effect on the production and have influence on the burning properties of the final product, too. As result, 8 critical parameters of raw materials were defined:

- ash-,
- cellulose-,
- carbohydrate-,
- dry matter-,
- extract-,
- hemicellulose-,
- lignin-, as well as,
- moisture content.

The *ash content* connects the non-burnable part of the raw material. The by-product of consumption is the ash, and minimum quantity is one of the most expected requirements. The melting point of ash is an important parameter in many aspects, too.

The calorific values can be characterized by proportion of burnable material to the moisture content of the raw material. The *carbohydrate-, the cellulose- and the hemicellulose* content can provide information about burnable part of raw material, and they can forecast the amount of ash, too [35]. In addition, the cellulose and the

hemicellulose play important role in bonding development, they have effect on quality of mechanical bonds, too [36].

The *dry matter* content probably the most important feature during the pellet production, and also party at usage, too [3].

Extract components inside the biomass materials typically have high calorific value and can play a role or can inhibit the establishment of different bonds, thereby they affect the physical and quality parameters of the finished products [34].

Lignin is a biological binder. Proper quantity of moisture and temperature are necessary for its activation. It has a lubricant function, too, and has effect on friction coefficient, moreover, on the properties of the final product, too [33].

The *moisture content* is one of most important parameter of biomaterials. It can act on friction coefficient, responsible for bond development, and properties of final product [32].

4.2. Evaluation of the measurement methods

The paper proposes a classification for the raw material measurement methods by introduction of three evaluation coefficients (device-, time requirement, and degree of complexity):

- The *device requirement* was measured by the number and features of applied equipment, like materials, devices, and their estimated costs.
- The *time requirement* was estimated by measurement time of the method, with the sample preparation time, and waiting time if it is necessary.
- The *degree of complexity* was determined by difficulty, multiplicity and circumstantiality of the measuring method.

This is a new measurement applicability index, which can be between 1 and 1000, the best index is 1, and the least favourable is 1000, so, small values represent more efficient measuring methods. All three test coefficients were determined by the authors for all collected, possible measuring methods. The multiplication of these three values result the final score of the individual solutions. The authors applied "simple" the multiplication of the individual factors (device-, time requirement and complexity). However according to their experiences, ability, know-how and/or further strategical capabilities the individual companies they can give multiplicative weights to the individual factors in order to personalise their favourable solution order. On the other hand, if a sophisticated financial controlling solution would have been given, these three aspects could be brought to a unified basis through the calculation of device, time and degree of complexity to cost. However, typically such a reliable and precise controlling solution is not available globally; only company specific, individual solutions can be applied. Finally, the reported evaluation does not change the sequence of the proposed measuring method for most of the analysed pellet quality measures in a relative wide range of such weightings, since the index values of the best solutions are not too close to each-other.

In order to have a more exact evaluation methodology the following applicability index calculation methodologies are proposed:

Device requirement: To calculate the device requirement index, the number of needed equipment was counted, e.g. on the basis of the related standards' descriptions (not including the number of basic laboratory aids - such as a pliers, a test tube, etc.) and the purchase value of the required assets were summarized. Both aspects are indexed according to Table 2. and the final device requirement coefficient is the rounded average value of these two indexes (price and amount).

Table 2.
Indexing the price and the amount of required measuring equipment

Price of required equipment			Amount of required equipment		
From-	-To	In-dex	From-	-To	In-dex
	≤ 3800 USD	1		1 pcs	1
> 3800 USD	≤ 4800 USD	2	2 pcs	3 pcs	2
> 4800 USD	≤ 5700 USD	3	4 pcs	5 pcs	3
> 5700 USD	≤ 6700 USD	4	6 pcs	7 pcs	4
> 6700 USD	≤ 7700 USD	5	8 pcs	9 pcs	5
> 7700 USD	≤ 8600 USD	6	10 pcs	11 pcs	6
> 8600 USD	≤ 9600 USD	7	12 pcs	13 pcs	7
> 9600 USD	≤ 10500 USD	8	14 pcs	15 pcs	8
> 10500 USD	≤ 11500 USD	9	16 pcs	17 pcs	9
> 11500 USD		10	> 18 pcs		10

Time requirement: The time index is calculated according to the Table 3. For determining the time requirement, e.g. according to the related standards, each component of the measurement process is considered, e.g. test piece preparation and handling, too.

Table 3.
Indexing the time requirement of the methods

Time requirement of method		
From-	-To	Index
	≤ 1 h	1
> 1 h	≤ 3 h	2
> 3 h	≤ 5 h	3
> 5 h	≤ 10 h	4
> 10 h	≤ 15 h	5
> 15 h	≤ 20 h	6
> 20 h	≤ 25 h	7
> 25 h	≤ 30 h	8
> 30 h	≤ 50 h	9
> 50 h		10

Degree of complexity: For determining the degree of complexity the number of required measuring process steps and their difficulties concerning users' know-how is considered. In the proposed methodology the individual measuring methods were compared to each other and the simplest one received the index one and the most complex the index ten.

These evaluation methodologies resulted in a numerical assessment and ordering of the individual pellet qualification and measurement techniques into the index range between one and ten and the final score is the multiplicative value of the three indexes.

The examined methods are able to determine the structural components of the biomass, the lignin and the extracts, as well as the moisture, dry matter and ash content. Several methods were examined within the same method-group, and the applicability of the methods showed high variance in most of the cases, as shown in Fig. 3.

Based on applicability index, there are more favourable and less favourable methods for measuring the same parameter ordered into a method-group.

4.3. Ranking of the measurement methods

In Table 4 the reviewed measurement methods are presented, grouped by the evaluated, critical raw material parameters.

Determination of *ash* content has more outstanding methods. *AOAC 942.05 method has the lowest applicability index (meaning that this is the best solution)*. This procedure is mainly recommended for the determination of ash content in feed, but according to the standard recommendation for biomass materials, too.

Less favourable methods are available for determination of *carbohydrate* content. There is no significant difference between the tested methods and the *best practice (ASTM E1758-01)* has an index value of 810.

For the *cellulose* content, the *Kürschner-Hoffer method is the most optimal*. During this process the wood/raw material is treated with a mixture of nitric acid-ethanol. As next, Lignin is nitrated and partially oxidized so, that it coalesces with simultaneous dehydrated hemicellulose.

Determination of the *dry matter* content, the *NREL/TP-510-42621-2* offers the best alternative.

Some *extract* components of biomass materials are water-soluble, but other, bigger sized parts of the extracts are soluble in organic solvent. There are methods with low applicability index, which are suitable for the water soluble extracts evaluation. The efficiency of the process can be improved with higher temperature of water, so *the most favourable method for water-soluble extract determination is the „Hot water-soluble extract” method*. Measurement of the quantity of organic solvent soluble extract is more complicated and has higher equipment and time requirements. During the measurement, the extracts of biomass are determined with organic solvent, then their quantity are determined by extra treatment of the residual material. Among the investigated procedures, the most favourable for this task is the *„Organic solvent-soluble extract” method*.

There are not significant variances between *holocellulose* determination methods, but based on applicability index, the most favourable method is the *Wise-method*, which is carried out in acetic acid medium by oxidation with sodium chlorite.

The *Klason method* is the most optimal to determination of *lignin* content, although this process also has a great need for equipment and complexity.

Among the examined parameters, the *moisture* content determination methods are the most auspicious, fitting easily into the daily production. The *two, top ranked methods (Moisture Meter Spec., ASTM E1358-97)* use automatic moisture measurement, which determine the moisture content based-on conductivity.

There are promising methods for the moisture, the dry matter, the ash and for the extract content measurement, among the overviewed measurement methods; however, the solutions for measuring of holocellulose, cellulose, structural carbohydrates, and lignin content are more complicated (their applicability index is relative poor/high). Beyond finding the most suitable measurement methods the optimal allocation along the pellet material flow has to be determined, too, from practical point of view of pellet production.

Table 4.

Analytical methods evaluation for pellets' raw materials measurement based on complexity, device and time demand

Analytical methods	Degree of complexity [1-10]	Device requirement [1-10]	Time requirement [1-10]	Applicability index
Determination of ash content				
AOAC 942.05	2	4	2	16
ASTM E1534-93	3	3	4	36
ASTM D1102-84	2	8	4	64
ASTM E1755-01	4	7	9	252
NREL/TP-510-42622	5	8	9	360
Determination of carbohydrates				
ASTM E1758-01	9	10	9	810
NREL/TP-510-42618	9	10	10	900
ASTM E1821-08	10	10	10	1000
Determination of cellulose				
Kürschner-Hoffer method	3	4	4	48
Normann-Jenkins- method	7	6	5	210
Wise method	7	7	6	294
Gross-Brau method	6	6	9	324
Determination of dry matter content				
NREL/TP-510-42621-2	2	3	4	24
NREL/TP-510-42621-1	3	2	5	30
ASTM E1756-08-2	2	4	4	32
ASTM E1756-08-1	3	7	9	189
Determination of extract				
Hot water-soluble extract	2	3	4	24
Cold water-soluble extract	1	4	10	40
ASTM E872-82	4	4	3	48
Organic solvent-soluble extract	5	7	9	315
ASTM E1690-08	9	7	10	630
NREL/TP-510-42619-2	9	8	10	720
NREL/TP-510-42619-1	9	8	10	720
Determination of holocellulose				
Wise method II.	7	5	5	175
Chlorination method	7	6	5	210
Jayme method	7	5	9	315
Determination of lignin				
Klason method	7	7	4	196
Halse method	7	7	8	392
ASTM E1721-01	10	9	9	810
Determination of moisture content				
Moisture Meter Spec.	1	1	1	1
ASTM E1358-97	1	1	1	1
Automatic drier	1	1	2	2
Xylene distillation	3	2	2	12
Dry to constant weight	2	2	3	12
ASTM E871-82	3	3	7	63

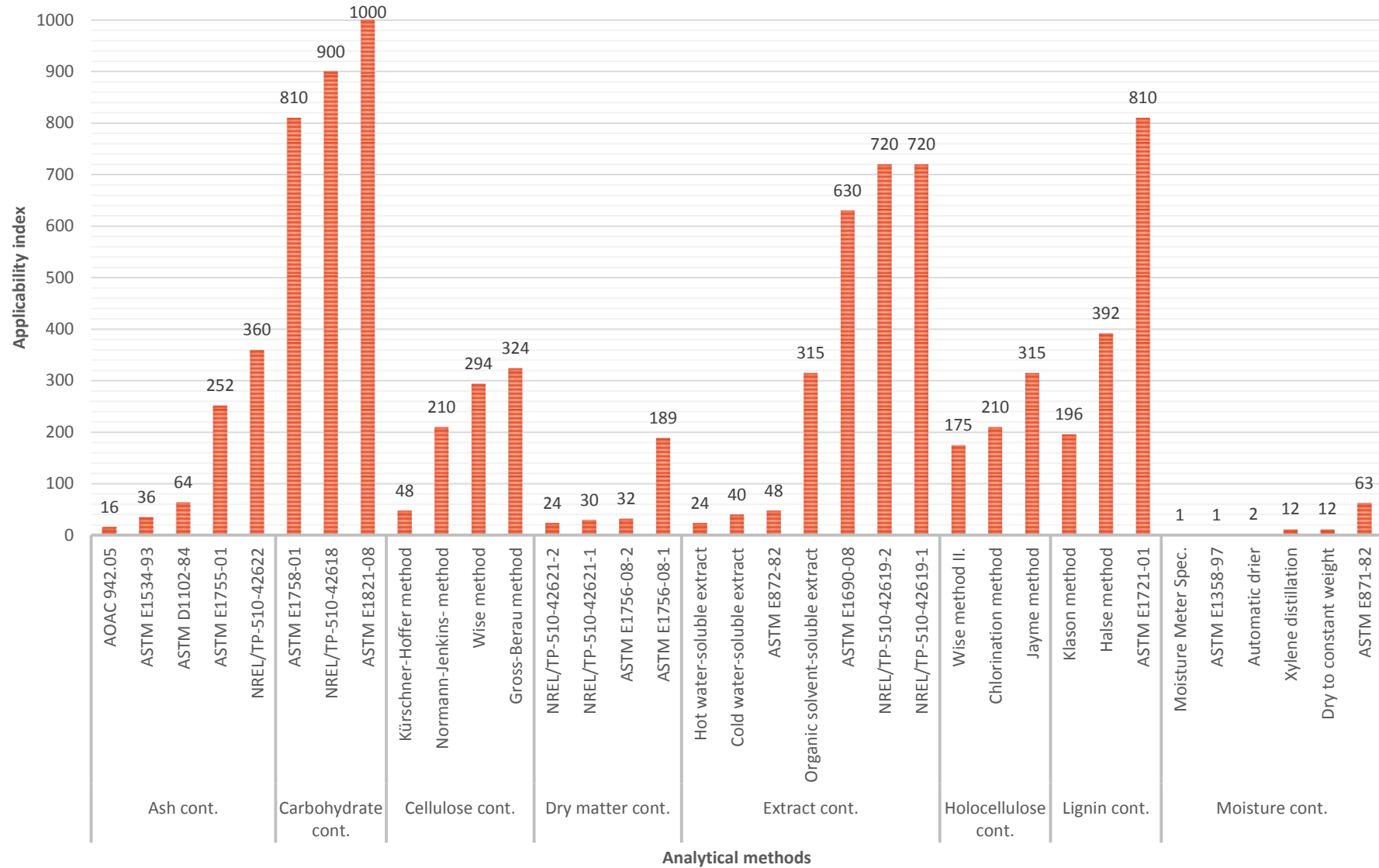


Fig. 3. Raw material measurement methods ranking for pellet production

5. Positioning of the proposed pellet raw material measurement methods along the material flow

Fig. 4. shows the possible, rational positions of the most promising measurement methods along the pellet production process.

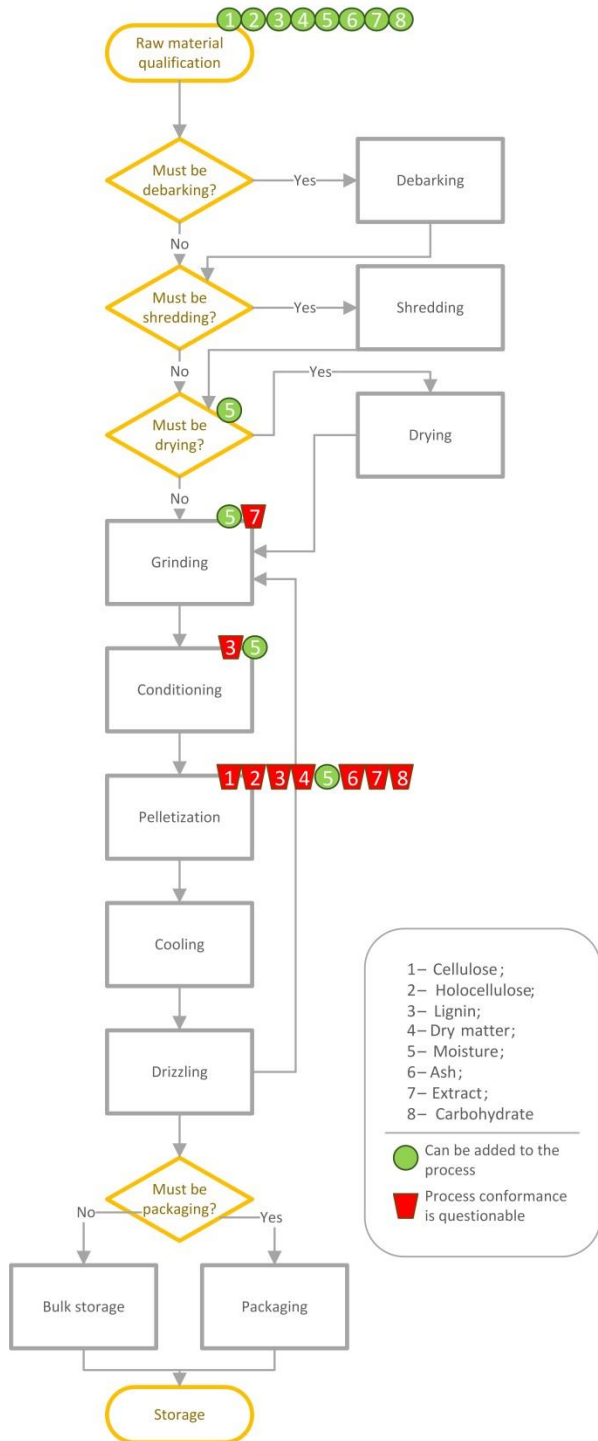


Fig. 4. Pellet production process with possible positions of measurement methods

All of the analysed methods can be added to any point of the process theoretically, but the practical implementation can raise many questions. There are just few fully automated measurement methods for these parameters, while many others are typically highly complex and requires lots of human resources- and time efforts. In Fig. 4. the theoretical and also the proposed practical positions are appointed with separate colours and forms. The green positions highlight the optimal allocation of the selected measuring methods considering many practical aspects into the account. Due to the previously mentioned limiting factors (complexity, human resource and time requirements), most of the measurement methods can be effectively integrated into the process only in the material qualification at the beginning of the material handling cycle. According to the measurements positioned in Fig. 4., all of the key quality factors of pellets are controlled similar to the qualification of the final product. So, one of the main differences between the proposed methodology and the current regulation status (on the ENplus standard basis) is that the measurements are done earlier in the production stages. This has many advantages, e.g. it enables significantly higher level production control and gives the ability to the manufacturers producing pellets not only from the classical wood but from other resources like agripellets having typically high distribution in their raw material characteristics. Nowadays producers solves this issue differently in their individual, personalized ways, e.g. since the ENPlus standard does not give recommendation and support for such challenges, only a categorization is given based on possible application types (industrial or non-industrial application), so, the paper goes beyond the current approach.

6. Conclusions

The growing market of pellets for energy production requires applying new biomass raw materials beyond the traditional wood. In the case of the pellet product the quality of the raw materials is a crucial point that is not controlled in the today's standards on the appropriate concernment. Pelletability and combustion properties of solid biogenic raw materials are determined by

- their molecular structure
- and their elemental composition.

The mixes of wood raw materials have low variability, but in case of non-woody raw materials the variability is high, it is especially important to define critical factors of these materials, because high-quality biofuels, that can satisfy the requirements, can be produced only with controlled and optimized raw material parameters and production processes. In spite of this, the today's quality standards do not include the raw material qualification appropriately. The origin of the raw material is the only controlled parameter for the finished wooden pellets classification. Consequently, the quality tools have to be supplemented with rules and recommendations about

material quality and qualification, too, this is the main aim of the paper.

A broad range of the related, possible and available raw material measuring methods was examined. These methods are suitable to determine the structural component of the biomass, the lignin and the extracts, as well as the moisture, dry matter and ash content. For finding optimal solutions for the raw material evaluation, a novel applicability index was proposed in the paper and estimated for all analysed methods applying the following three test coefficients:

- complexity of the method.
- device requirement of the method,
- and time requirement of the method.

The obtained applicability index is able to rank the individual measuring solutions within the evaluated parameter-group, too. The analysed methods showed high variance according to applicability within parameter-group, and based-on applicability index, there are favourable and less favourable methods. There are promising methods to the moisture, to the dry matter and to the extract content determination, among the overviewed measurement methods, however, the solutions for measuring of holocellulose, cellulose, structural carbohydrates, lignin and ash content are more difficult (since, their applicability index is relative poor/high), moreover, their integration into pellet production cycle is more complicated.

The paper also appointed theoretically possible measurement positions for the selected, available best methods and suggested their optimal allocation along the material and production flow.

Considering further research activities, the today's available, introduced pellet standards will be supplemented with harmonized recommendations according to the result of the reported research.

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