THE USE OF A NON-INVASIVE ELECTRICAL METHOD TO ASSESS THE CHEMICAL COMPOSITION, HARDNESS, AND COLOR OF DURUM WHEAT GRAIN CULTIVATED IN AN INTEGRATED SYSTEM

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Abstract

The aim of this research was: (1) to assess the effect of an integrated cultivation system of native durum wheat (6 variants), differentiated by a nitrogen fertilization dose (0, 80, 120 kg·ha^{-1}) and growth regulator use (Medax 350 SC, with growth regulator – GR, without growth regulator – WGR) on changes in selected quality parameters (protein, lipid, and starch contents; hardness; color) and electrical parameters (conductive – Z and capacitive – C_p, C_s) of the grain; and (2) to determine the correlations between the examined quality traits and electrical properties of the grain to indicate the possibility of using a non-invasive electrical method to assess grain quality.

The grain of durum wheat cultivated in the six variants tested differed mainly in the starch and lipid contents, which indicates the feasibility of tailored optimization of grain production. The highest contents of starch and total lipids, and the highest grain hardness were obtained in the cultivation variants GR+0N and WGR+0N, whereas the highest protein content – upon wheat fertilization with 120N. The study demonstrated a different strength of the correlations between the tested parameters depending on the cultivation method (WGR-GR; N: 0, 80, 120 kg·ha^{-1}). In the WGR+0N variant (environmentally friendly), the strongest correlation between grain quality traits and electrical properties was obtained for both the conductive (Z) and capacitive (C_p, C_s) parameters in the entire analyzed range of current frequencies. The GR application during cultivation hampers the possibility of using electrical parameters to assess grain quality. In turn, the quality of the grain produced WGR can be effectively assessed using the Z, C_p, and C_s parameters in the entire measuring frequency range (1kHz-1MHz), while in the variant with GR, only impedance (Z), measured at high frequencies (100kHz, 1MHz) can be used.

Keywords: durum wheat grain; agrotechnical parameters; quality evaluation; electrical method, impedance, electric capacity

Introduction

Durum wheat (*Triticum durum* Desf.) occupies about 8% of the global acreage intended for wheat cultivation, and its production accounts for approximately 5% of the global wheat harvest (Colasuonno et al. 2019). It is cultivated in a dry and hot climate, where its grain yield is diminished by water shortage. Spring durum wheat is mainly grown worldwide, as it is the oldest form of this cereal. Winter forms, that had been developed from the spring forms, are cultivated in the South-Eastern Europe. After 1960, durum wheat cultivation shifted from the Mediterranean region more to the north of Europe. This was due to significant breeding progress and registration of varieties in such countries as Austria, Germany, Hungary, Czech Republic and Poland (Banach et al. 2021; Alemu, 2017; Kabbaj et al. 2017).

Due to its exceptional technological properties (hard endosperm and high contents of gluten and carotenoids) and nutritional value, durum wheat grain is used to make top-quality pasta (Morris et al. 2015, Xiao Fu et al. 2018, Colasuonno et al. 2019).

Durum wheat species may gain in importance in Poland due to the ongoing climate changes coupled with breeding progress in its cultivation and growing interest expressed by the food industry (Polish National List of the Research Center for Cultivar Testing 2021). The cultivation of this demanding wheat species and its grain yield are largely affected by weather conditions (Żuk-Gołaszewska et al. 2016, Semenov et al. 2014). Despite these impediments, breeding and agro-engineering works are undertaken regarding different varieties of durum wheat to produce the most optimal ones for the north-eastern Europe (Żuk-Gołaszewska et al. 2016, Polish National List of the Research Center for Cultivar Testing 2021). So far, a few winter and spring durum wheat varieties have been introduced into the Polish National List of Agricultural Plant Varieties, these being: Komnata, Ceres, SM Metis, SM Eris, SM Tetyda as well as SMH 87, respectively (Polish National List of Agricultural Plant Varieties 2018, Polish National List of the Research Center for Cultivar Testing 2019, 2020). An additional course of action is the production of plants in an integrated system, the aim of which is to draw from the potential of sustainable economic, ecological, and social development. The exploitation of the first two potentials is feasible through the conscious use of innovative technologies and manufacturing techniques, continuous improvement of environmentally friendly management practices, as well as implementation of biological progress. The production of high-quality cereal grains and grain yield size are expected to be ensured by, among others, agro-engineering factors, including the level of nitrogen fertilization and the use of growth regulators (Banach et al. 2021).

The development of automation in integrated agricultural production has spurred the interest in the electrical properties of grain and cereal products. In addition to using them in electro-engineering, as an alternative to the conventional methods for process monitoring and analysis in storage and processing practice (Łuczycka 2009; Nelson & Bartley 2000, Nelson & Trabelsi 2017), research is also carried out on the possibility of replacing analytical methods of grain quality assessment with electrical methods. However, the complex morphological structure of kernels, chemical composition (protein, lipids, starch, minerals), water content (in cells, intercellular spaces, and membranes), and the lack of repeatability of dimensions and other physical features require in-depth research in this area. Our previous research addressing the use of an RCC substitute model of food products showed that the electrical parameters, i.e. impedance and admittance, can be used to identify the variety, geometric features, and grain moisture content (Majewska et al. 2008); to distinguish cereal species (wheat, barley, rye, triticale, oat) and varieties (Banach et al. 2017); and to determine the extent of barley grain damage (Banach et al. 2020). Promising results concerning the use of electrical features of grain/seed products for their quality assessment were also achieved in the case soybean (Jha et al. 2011), maize (Bansal et al. 2015), and wheat flours (Łuczycka et al. 2013).

Summing up, it needs to be stated that a global trend can be observed regarding the increasing consumption of products made of durum wheat grain, and also that the use of integrated agricultural practices of cereal grain cultivation is conducive to modeling the quality of their production, and finally that the need to develop a quick method for assessing the quality characteristics of grain using environmentally friendly electrical methods is a current challenge for scientists. Therefore, a study was undertaken that aimed at:

- ✓ evaluating the effect of an integrated cultivation system, differing in a nitrogen fertilizer dose and growth regulator use (with/without, GR/WHR) on changes in the selected quality traits (protein, lipid, and starch contents; hardness; color) and electrical properties (conductive and capacitive) of the grain of native durum wheat;
- ✓ determining correlations between the analyzed quality traits and electrical properties of durum wheat grain to identify the feasibility of employing a non-invasive electrical method for grain quality assessment.

Material and methods

➢ ***Cultivation of durum wheat in an integrated system***

The experimental material included grain of durum wheat var. SMH 87 derived from a field experiment established in the north-eastern Poland (53°40'N, 19°50'E) – harvest 2016. Durum wheat cultivation in an integrated system (6 variants) was differentiated by a nitrogen fertilization rate and use of a growth regulator Medax 350 S.C. (with/without growth regulator; GR/WGR) expected to prevent lodging (preventive action). Nitrogen was applied in three doses: no fertilization (0N), 80 $kg \cdot ha^{-1}$ (80N), and 120 $kg \cdot ha^{-1}$ (120N). A detailed description of the fertilization scheme was provided in our previous research (Banach et al. 2021). After harvest, grain was cleaned of physical impurities and seed coat residues. Ca. 500 g of the grain were collected to glass bottles from each cultivation variant, and the bottles were stored in the climatic chamber (Memmert, USA) at air temperature of 20±0.1°C. The grain intended for research had a standardized moisture content of 14±0.5% (PN-EN ISO 712:2012).

➢ ***Determination of the selected chemical components of the grain***

The grain comminuted with a WZ-1 laboratory grinder (Sadkiewicz Instruments, Poland) was determined for the contents of: total protein – with the Kjeldahl’s method (PN-EN ISO 20483:

2014-02), total lipids – with the Soxhlet's method, and total starch – with the polarimetric method acc. to the methodology described by Krełowska-Kułas (1993). Determinations were performed in 6 replications for each component, and the results were expressed per % of dry matter (d.m.).

- ***Hardness measurements***

Hardness of single kernels placed furrow-down on a table was measured using a Universal Testing Device (Instron 5942; Instron Corp., USA). A uniaxial compression test (flat-truncated cone, Ø=12.6 mm; speed of the working head, 10 mm/min) was performed to measure the maximum compression force (F_{max}) needed to reach 50% grain deformation, in 16 replications for each of the 6 grain samples from cultivation variants tested. The results of the tests were analyzed using the computer software Bluehill II.

- ***Color measurements***

Color measurements of the grain were made using a Hunter Miniscan XE Plus colorimeter (HunterLab, USA), set to collect spectral data with illuminant A/observer D65/10°. Before the measurements, the colorimeter was calibrated using a black and white ceramic plate. Grain color was determined in the CIELab scale, measuring lightness (L*), redness (a*), and yellowness (b*), in 12 replications.

- ***Electrical measurements***

Electrical measurements were performed using a measuring system composed of the following devices (Fig. 1):

- a measuring sensor – a glass container (internal dimensions: L: 88.80 × D: 49.20 × H: 76.20 mm) with flat electrodes made of acid-proof steel having the surface area of ca. 68 cm^2, attached adjacent to two opposite longer container's walls,
- a metal container with a water jacket connected to a thermostat (PolyScience, USA),
- an LCR meter, type E4980a (Agilent, USA) with a measuring attachment.

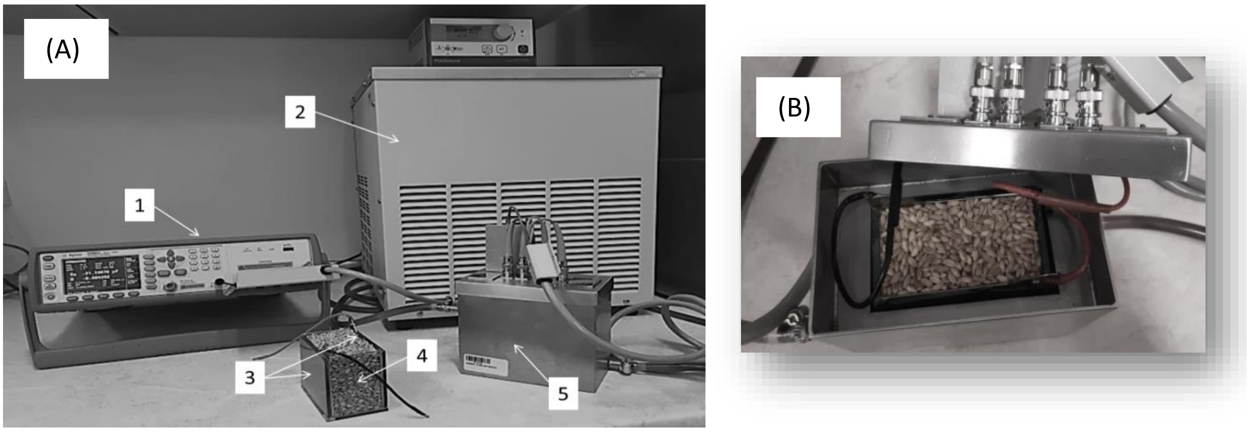

Fig 1. The measurement system used for electrical properties examination of the wheat grain (A): 1- LCR meter, type E4980a 2- thermostat; 3 – measuring glass cell with plate electrodes, 4- examined wheat; 5- water jacket; (B) the internal view of the measuring cell with sample, water jacket and connecting cables

Before measurements, the experimental material with a standardized moisture content of 14±0.5% was stored (temp. 20±0.1˚C/24h) in tightly closed glass bottles in a climatic chamber (Memmert ICP500, Germany). The LCR meter with wires was calibrated. The glass container was filled up with grain (ca. 250g) and placed in the metal container with a water jacket that was next thermostated to a temperature of 20±0.05 °C (Fig 1A). The wires fixed to the electrodes of the measuring sensor were connected to the wires of meter's attachment, mounted in the upper section of the thermostated system (Fig 1B), and measurements were performed. The electrical properties of the grain were determined based on the measurements of conductive parameters (impedance – Z) and capacitive parameters (C_p and C_s), performed at frequencies (f) of 1kHz, 100kHz, and 1MHz, and voltage of 200 mV. The measuring frequencies were selected based on the results of our previous investigations (Majewska et al. 2008, Banach et al. 2020). The electrical measurements were conducted in 12 replications for each of the 6 grain samples using an RCC substitute model of food products (serial-parallel resistance + capacitance) – Fig 2, which was also employed to determine change in the quality of animal and plant products in our earlier research (Banach et al.

2012, 2017, 2021; Banach & Żywica 2021, Żywica et al. 2012), and results were registered in an external memory of the meter.

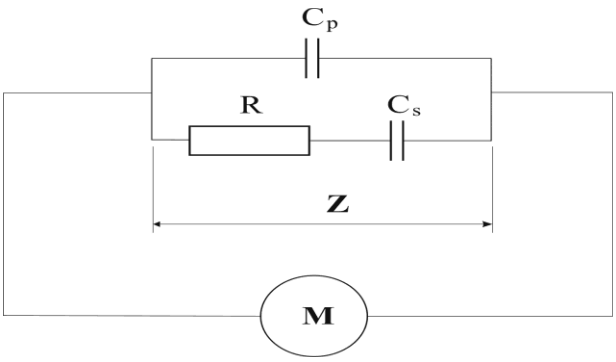

Fig 2. RCC equivalent model for measuring of electrical properties food products (Z - impedance; R - resistance; C_s - series equivalent capacitance; C_p - parallel equivalent capacitance; M - measuring device)

➢ *Statistical analysis*

The results of measurements were processed statistically using Statistica 13.3 software (StatSoft Inc., Tulsa, OK, USA). Data was analyzed employing procedures which enabled: (1) determining basic statistical measures (mean values ± standard deviation), (2) comparing the values of the analyzed traits of the grain produced using different agro-engineering factors, and (3) determining correlations between the analyzed physicochemical and electrical properties of the grain. The computations were performed at significance levels of $p<0.05$, $p <0.01$, and $p<0.001$. Furthermore, the Principal Component Analysis (PCA) was conducted to identify correlations between the quality traits and electrical properties of the grain of durum wheat grown without (WGR) and with the application a growth regulator (GR), on a two-dimensional plane (Ringnér, 2008, Abdi and Williams, 2010).

Results and discussion

Changes in the physicochemical properties (technological) of the grain

Starch is the major storage polysaccharide accumulated in wheat grain, composed of two fractions – linear amylose and branched-chain amylopectin. Starch granules are spherocrystalline and their size is species-dependent (in wheat, it ranges from 10 to 24 μm), whereas their density depends on the hydration degree. They feature double refraction and are coated with a thin lipoprotein layer (Gąsiorowski 2004). Starch of durum wheat grain is strongly bound to proteins of the endosperm, which results in its glassy and compact structure. In turn, a less compact structure with numerous open spaces and physically discontinuous protein matrix is observed for starchy kernels (Xiao Fu et al. 2018).

The starch content of durum wheat grain from particular variants of integrated cultivation ranged from 60.9 to 65.1 % d.m. (Tab. 1). It was lower from the average values reported by Lafiandra et al. (2014) for ripe wheat grain, i.e.65 - 75%, but fitted the range expected by Gąsiorowski (2004), i.e. 55 - 70 %. Wheat cultivation in the tested variants differentiated starch content of the grain. The highest starch content was determined in the grain from the variant with the growth regulator and without nitrogen fertilization (GR+0N). With nitrogen dose increase (N: 80 and 120 kg·ha^{-1}), the starch content of the grain decreased significantly ($p<0.01$). In turn, wheat cultivation in variants GR+80N and GR+120N resulted in a significant ($p<0.01$) increase in the content of this chemical component (Tab. 1).

Lipids represent a large group of chemical compounds shearing the following common traits: solubility in organic solvents and insolubility in water. They are a quantitatively minor but crucial component of wheat grain. The total lipid content of wheat grain ranges from ca. 0.9 to 3.3 %; however, the highest average content (2.8 % d.m.) can be found in durum wheat grain. The lipid content is positively correlated with kernel hardness and color. Durum wheat varieties have higher contents of both total lipids and polar lipids (glycolipids and phospholipids) compared to soft wheats (Gąsiorowski 2004). The total lipid content of the grain analyzed in the present study

Table 1. Changes in the physicochemical properties (starch, lipid and protein content; values F_{max} - maximum compression force) of the durum wheat grain depending on growth regulator application (WGR, GR) and nitrogen fertilization (0, 80, 120 kg·ha^{-1}).

Parameters	Without the application growth regulator (WGR)			With the application growth regulator (GR)			Significant differences WGR - GR		
	Fertilization [kg·ha^{-1}]								
	0N	80N	120N	0N	80N	120N	0N	80N	120N
Starch [% s.m]	64.62±0.27^{a}	62.01±0.32^{b}	60.90±0.24^{c}	65.12±0.34^{a}	63.44±0.28^{b}	61.85±0.05^{c}	NS	p<0.01	p<0.01
Lipid [% s.m]	2.43±0.05^{aA}	2.31±0.01^{b}	2.22±0.09^{bB}	2.51±0.01^{a}	2.11±0.05^{b}	2.24±0.03^{c}	p<0.05	p<0.01	NS
Protein [% s.m]	12.717±0.530^{a}	14.157±0.388^{b}	14.425±0.382^{b}	11.450±0.418^{a}	14.022±0.504^{bB}	15.825±0.851^{Cd}	0.01	NS	NS
F_{max} [N]	131.87±17.98^{a}	126.47±10.66^{a}	122.87±19.88^{a}	120.90±9.08^{a}	120.51±14.22^{a}	134.38±9.57^{b}	NS	NS	NS
L*	53.92±0.54^{a}	53.27±0.79^{a}	52.11±0.64^{a}	53.49±1.04^{a}	52.93±0.48^{a}	52.84±0.79^{a}	NS	NS	NS
a*	8.78±0.72^{a}	8.92±0.55^{a}	8.88±0.21^{a}	8.67±0.71^{a}	8.82±0.60^{a}	8.69±0.64^{a}	NS	NS	NS
b*	28.42±0.51^{a}	28.39±0.57^{a}	27.91±0.54^{a}	28.14±0.63^{a}	27.35±0.74^{a}	27.87±0.62^{a}	NS	NS	NS

NS – no significant differences

$^{a-d}$ – mean values in columns indicated with various small letters are statistically significantly different at p<0.01;

$^{A-B}$ – mean values in columns indicated with various capital letter are statistically significantly different at p<0.05

$^{a-a}$ – mean values in columns indicated with the same small letters non statistically differences ranged from 2.1 to 2.5% d.m., with the highest content determined in the grain from the variant without nitrogen fertilization and with growth regulator (GR). The total content of lipids was more significantly affected by nitrogen fertilization. The analysis of the effect of growth regulator use (WGR-GR) showed that the cultivation variant had a significant ($p<0.01$; $p<0.05$) effect on lipid content of the grain from the variants with nitrogen fertilization at 0 and 80 $kg\cdot ha^{-1}$ (Tab. 1).

The total protein content of the grain (8-18 %) defines the functionality and processing options of different wheat species and types. The main storage proteins of wheat grain, being at the same the key indicators of its technological quality, include gluten proteins originally divided into gliadins and glutenins (prolamines and glutelins). Durum wheat grain has a high total protein content (11.5-18.5 % on average) and is characterized by different proportions of individual protein fractions compared to the common wheat grain. The content of the gliadin protein fraction is higher than that of the high-molecular fraction of glutenin proteins. Owing to this, dough made of semolina is more plastic than elastic, which determines the high usability of durum wheat grain in the pasta making industry (Gąsiorowski 2004, De Santis et al. 2017, Dragov 2021).

The total protein content of the analyzed grain ranged from ca. 11.5 to 15.8 % d.m. It was most beneficially influenced by the cultivation variant GR+120N and the least beneficially by the GR+0N variant (Tab. 1). Also Obuchowski et al. (2007) reported a beneficial effect of nitrogen fertilization (at 100 - 150 $kg\cdot ha^{-1}$) on the total protein content of the grain of 4 durum wheat varieties grown under Polish climate conditions. The contemporary durum wheat varieties require higher rates of nitrogen fertilization (up to 100 - 120 $kg\cdot ha^{-1}$) than the old varieties to reach their maximum grain yield. Certain cultivars need even higher fertilization doses to increase their grain protein percentages; however, these agricultural strategies are economically ineffective (Giuliani et al. 2011, Mefleh et al. 2018).

The hardness of wheat grain is the most important indicator of its structural and mechanical properties, including especially endosperm features (Xiao Fu et al. 2018). This property defines the potential grain behavior during milling and affects such technological traits as: the extraction rate, fraction size, and water absorption of pasta flour/semolina, starch damage degree, and starch susceptibility to enzymatic processes. One of the measures of wheat kernel hardness is their resistance to compression to the assumed deformation.

Grain hardness, expressed as the maximum compression force (F_{max}) required for its 50% deformation, ranged from 120.5 to 134.4 N (Tab. 1). The analyzed durum wheat grain was harder than the grain of common wheat (68.0 - 109.5 N) and spelt grain (37.4 - 98.8 N) tested by Żuk-Gołaszewska et al. (2018). These differences point to the better cohesiveness of starch granules with protein and a lower number of free air compartments, which ultimately indicates potentially higher resistance of the analyzed durum wheat grain during milling (Morris et al. 2015, Xiao Fu et al. 2018). Significant and beneficial changes in hardness values were noted in the cultivation variant GR+120N (Tab. 1).

Grain color is another important technological trait of wheat. The yellow-amber color of semolina is caused by the carotenoid yellow pigment content in the entire grain. The high content of carotenoid pigments is especially valued in the case of durum wheat intended for top-quality semolina production as it determines pasta color. It is positively correlated with color parameter b* (yellowness) in the CIE system (Żuk-Gołaszewska et al. 2018, Colasuonno et al. 2019). Grain color, expressed by color parameters L* (52.1-53.9), a* (8.7-8.9), and b* (27.4-28.4) did not change significantly across the wheat cultivation variants (Tab. 1). Similar results were obtained by Obuchowski et al. (2007) who analyzed the effect of durum wheat fertilization with nitrogen on changes in color parameters (L*, a*, b*) of pasta flour produced from its grain. Low variation (less than 10%) of color parameters of common wheat and spelt grain, cultivated in organic farming system for 3 years, was also reported by Żuk-Gołaszewska et al. (2018).

Changes in the electrical parameters of the grain

Among the multiple physical properties, the electrical properties of cereal grain enable the non-invasive identification of its quality traits, including structural changes of the grain, like e.g. damage degree (Banach et al. 2020). However, due to heterogeneity, cellular structure, chemical composition, and shape of various biological materials tested, a specific measurement technique should be developed for each material type (Łuczycka 2009).

Like in the case of selected chemical components determined in this study, measurements of the electrical parameters (Z, C_p, and C_s) of durum wheat grain demonstrated a significant effect of agro-engineering factors (WGR-GR; N: 0-120 kg·ha^{-1}) on changes in their values. These changes were additionally affected by the tested frequencies of the measuring current (1kHz-1 MHz).

Due to its cellular structure and chemical composition, cereal grain exhibits conductive and dielectric (capacitive) properties. In the process of electric current conduction, the cytoplasm exhibits the characteristics of a complex electrolyte (suspension), composed of proteins and amino acids, nucleic acids, lipids, and carbohydrates as well as low-molecular-weight organic compounds, mineral salts, and water. The electrical conductivity of the cytoplasm depends on the type, concentration, and mobility of ions (Żywica 2010). Given the above, measurements were made for one of the conductive parameters – impedance (Z), which is defined as complex resistance ($Z^2 = R^2 + (X_l - X_c)^2$). The grain of wheat cultivated without the growth regulator and with nitrogen fertilization at 80kg·ha^{-1} (WGR+80N) had significantly the highest impedance values (Z_{100kHz} = $3.36 \cdot 10^5$ Ω; Z_{1MHz} = $3.81 \cdot 10^4$ Ω), whereas the grain from the WGR+0N variant had the lowest Z values. The statistical analysis of the effect of the WHR-GR factor demonstrated significant ($p<0.01$; $p<0.05$) differences between Z values (measured at 1kHz 100kHz; 1MHz) of the grain produced with 0N and also between Z_{100kHz} values of the grain from the 120N variant (Tab.2).

These results point to a significant effect of protein content on the electrical conductivity and to the feasibility of producing grain with an increased protein content in the WGR+0N variant (environmentally-friendly, cost-effective) compared to the GR+0N variant. Increasing nitrogen fertilization rates (80-120N) in both the WGR and the GR variant caused no differences in the protein content of the grain, which is the main positive driver of grain yield and technological quality (Obuchowski et al. 2007; Giuliani et al. 2011, Mefleh et al. 2018). When investigating the

Table 2. Changes in the values (mean values ± standard deviation) of the electrical parameters (Z, Cp, Cs) durum wheat grain cultivated with the application (GR) and without application (WGR) of the growth regulator with different nitrogen fertilization (0, 80, 120 kg·ha^{-1}).

Frequency	Without the application growth regulator (WGR)			With the application growth regulator (GR)			Significant differences WGR - GR		
	Fertilization [kg·ha^{-1}]								
	0N	80N	120N	0N	80N	120N	0N	80N	120N
				Z [Ω]					
1 kHz	16.00±5.29·10^{6}a	14.50±1.09·10^{6}a	11.50±1.08·10^{6}b	12.50±1.76·10^{6}a	14.30±2.15·10^{6}bB	11.5±3.74·10^{6}Ca	p<0.05	NS	NS
100 kHz	1.65±0.15·10^{5}a	3.36±0.12·10^{5}b	2.87±0.05·10^{5}c	2.92±0.34·10^{5}a	3.27±0.12·10^{5}b	2.97±0.14·10^{5}ca	p<0.01	NS	p<0.05
1 MHz	2.85±0.50·10^{4}a	3.81±0.12*10^{4}b	3.32±0.12·10^{4}c	3.30±0.37*10^{4}a	3.70±0.15·10^{4}b	3.37±0.16·10^{4}ca	p<0.05	NS	NS
				Cp [F]					
1 kHz	1.19±0.05·10^{-11}a	1.06±0.09·10^{-11}b	1.32±0.13·10^{-11}c	1.23±0.19·10^{-11}a	1.07±0.17·10^{-11}b	1.21±0.09·10^{-11}ca	NS	NS	p<0.05
100 kHz	5.42±0.26·10^{-12}a	4.71±0.16·10^{-12}b	5.46±0.11·10^{-12}ca	5.47±0.68·10^{-12}a	4.84±0.19·10^{-12}b	5.33±0.26·10^{-12}ca	NS	NS	NS
1 MHz	4.83±0.23·10^{-12}a	4.18±0.12·10^{-12}b	4.78±0.17·10^{-12}ca	4.87±0.58·10^{-12}a	4.29±0.18·10^{-12}b	4.71±0.23·10^{-12}ca	NS	NS	NS
				Cs [F]					
1 kHz	1.32±0.06·10^{-11}a	1.15±0.09·10^{-11}b	1.46±0.15·10^{-11}c	1.37±0.21·10^{-11}a	1.17±0.02·10^{-11}b	1.33±0.11·10^{-}11ca	NS	NS	p<0.05
100 kHz	5.52±0.26·10^{-12}a	4.80±0.17·10^{-12}b	5.61±0.08·10^{-12}ca	5.57±0.71·10^{-12}a	4.93±0.18·10^{-12}b	5.41±0.26·10^{-12}ca	NS	NS	p<0.05
1 MHz	4.86±0.24·10^{-12}a	4.19±0.13·10^{-12}b	4.82±0.16·10^{-12}ca	4.90±0.58·10^{-12}a	4.32±0.18·10^{-12}b	4.71±0.21·10^{-12}ca	NS	NS	NS

NS – no significant differences

$^{a\text{-}c}$ – mean values in columns indicated with various small letters are statistically significantly different at p<0.01;

$^{A\text{-}C}$ – mean values in columns indicated with various capital letter are statistically significantly different at p<0.05

$^{a\text{-}a}$ – mean values in columns indicated with the same small letters non statistically differences effect of fertilization type in durum wheat cultivation, also Rossini et al. (2018) determined a lower protein content of the grain in the variant with organic fertilization compared to mineral fertilization; however, they did not notice a strong impact of this factor on protein and gluten percentage contents. Wheat grain yield and quality as well as nitrogen utilization effectiveness are influenced by environmental factors (Marinaccio et al. 2016, Di Mola et al. 2021). Our previous study (Banach et al. 2021) demonstrated that high-quality grain with desired flour quality parameters (flour extraction rate ≈ 64%; flour particle size ≈ 98%; L* ≈ 92) can be produced from spring durum wheat grown without the growth regulator and at 80 kg·ha^{-1} nitrogen fertilization. In addition, its results allowed concluding that this cultivation variant proved effective in reducing durum wheat production costs and diminishing natural environment pollution.

Solid state biophysics classifies cereal grain into the group of heterogeneous (layered) and weakly polarizable dielectrics, differing in mass. This is directly due to the high content of lipids in the cell wall and membrane. The size of the electric charge accumulated in the grain or the dipole moment is the factor that determines the behavior of the grain in the electric field. Therefore, the ability to accumulate an electric charge by durum wheat grains from the 6 cultivation variants was determined using capacitive parameters (strictly dependent on the geometric features, moisture content, chemical composition, and morphology of the grain).

The measurements of the capacitive parameters (C_p, C_s) of the grain showed different trends of changes than in the case of the Z values. In the WGR cultivation variant, significantly the highest C_p and C_s values were obtained at fertilization rates of 120N (f=1kHz, f=100kHz) and 0N (f=1MHz). In turn, wheat cultivation with GR contributed to the highest C_p and C_s values in the 0N variant (regardless of the measuring frequency). However, the statistical analysis of the effect of the WGR-GR factor demonstrated significant ($p<0.05$) differences only between the Cp_{1kHz} and Cs_{1kHz} and Cs_{100kHz} values of the grain from wheat fertilized with 120N (Tab. 2).

The analysis of correlations between the quality traits and electrical parameters of durum wheat grain produced without the growth regulator (WGR) showed that the nitrogen dose had a significant effect on the strength of these correlations (Tab. 3). Significant ($p<0.001$; $p<0.01$; $p<0.05$) correlations were found for impedance (Z) and capacitance parameters (C_p, C_s) of the grain produced in the WGR+0N variant, regardless of the measuring frequency. Increasing the fertilization rate to 80N impaired the possibility of using electrical parameters (Z, C_p, C_s) for grain yield and quality assessment only to these measured at the higher frequencies tested (f=100kHz and f=1MHz). At the highest nitrogen fertilization dose used in the study (120N), the strongest correlation was noted between Z_{1MHz} and $C_{p100kHz}$ and $C_{s100kHz}$ (Tab. 3). The viability of capacitance parameters in grain yield and quality assessment was also demonstrated by Cseresnyés et al. (2021) and Di Mola et al. (2021). The recommendations to use the electrical parameters only at higher frequencies of the measuring current (100 kHz, 1 MHz) may be due to the facts that the increasing frequency is accompanied by a successive decrease in the electric permeability value (current streams flow through the cell), that the capacitive resistance of the membrane practically contains the resistance of the lipid layer, and, finally, that the intracellular substance is involved in the conduction of electricity (Żywica 2010).

The statistical analysis of results from the wheat cultivation variant with GR demonstrated stronger correlations between quality traits and electrical parameters of the grain with increasing nitrogen rates, indicating especially the viability of C_p and C_s parameters in grain quality assessment (Tab. 4). In the case of wheat grown in the GR+0N variant, the most significant ($p<0.001$) correlation was demonstrated between grain chemical composition (protein, lipid, and starch contents) and impedance (Z) measured in the entire tested range of current frequencies. In turn, the values of C_p and C_s parameters were correlated only with lipid and starch contents. These correlations are also confirmed by the earlier described phenomena (WGR cultivation variant). The quality traits of the grain from wheat grown in the GR+80N variant correlated with the values of electrical parameters measured at the higher frequencies tested (Z_{100kHz}, C_s and $C_{p100kHz}$; C_s and C_{p1MHz}). The correlations demonstrated in the GR+120N variant were similar to those noted for the GR+80N variant; however, they more strongly indicated the possibility of using the C_s parameter (Tab. 4) to evaluate

Table 3. Analysis of the correlation between technological (lipid, protein, starch, hardness) and electrical (Z, Cs, Cs) parameters of hard wheat grain cultivated without retardant (WGR) and at different nitrogen dose (0, 80, 120 $kg \cdot ha^{-1}$)

Quality parameters	Z_{1kHz}	Z_{100kHz}	Z_{1MHz}	Cs_{1kHz}	Cs_{100kHz}	Cs_{1MHz}	Cp_{1kHz}	Cp_{100kHz}	Cp_{1MHz}
					WGR+0N				
Lipid [% s.m]	***	***	***	***	***	***	***	***	***
Protein [% s.m]	**	***	*	***	***	*	0.129	***	***
Starch [% s.m]	***	***	***	***	***	***	***	***	***
Hardness [N]	***	***	***	**	*	*	***	***	***
L*	**	**	**	0.734	0.943	0.763	**	**	**
a*	0.738	0.864	0.898	0.842	0.916	0.962	0.774	0.920	0.935
b*	*	*	*	0.218	0.235	0.249	*	*	*
					WGR+80N				
Lipid [% s.m]	0.515	***	***	0.945	**	***	0.462	***	***
Protein [% s.m]	0.319	***	***	0.994	***	***	0.953	***	***
Starch [% s.m]	0.138	***	***	0.914	***	***	0.165	***	***
Hardness [N]	0.349	*	*	0.457	*	*	**	*	*
L*	0.367	0.089	0.075	0.436	0.083	0.077	0.690	0.830	0.686
a*	0.289	0.718	0.727	0.257	0.729	0.714	0.871	0.996	0.960
b*	0.092	0.117	0.126	0.100	0.122	0.123	0.248	0.340	0.369
					WGR+120N				
Lipid [% s.m]	0.992	0.435	***	0.691	***	***	0.983	***	**
Protein [% s.m]	0.895	0.929	***	0.777	***	0.570	0.865	*	0.793
Starch [% s.m]	0.989	0.953	***	0.957	***	0.114	0.983	***	0.537
Hardness [N]	0.591	***	*	***	***	***	***	***	***
L*	0.770	0.965	0.651	0.171	0.845	0.854	0.301	0.145	0.089
a*	0.605	0.549	0.411	0.380	0.578	0.305	0.779	**	**
b*	0.700	0.916	0.796	0.988	0.975	0.974	0.928	0.780	0.819

*** - significance level $p<0.001$; ** - significance level $p<0.01$; * - significance level $p<0.05$

Table 4. Analysis of the correlation between technological and electrical parameters (Z, Cs, Cs, measured at the frequency of 1kHz, 100kHz, 1MHz) of hard wheat grain grown with retardant (GR) and differentiated nitrogen dose (0, 80, 120 kg·ha^{-1})

Quality parameters	Z_{1kHz}	Z_{100kHz}	Z_{1MHz}	Cs_{1kHz}	Cs_{100kHz}	Cs_{1MHz}	Cp_{1kHz}	Cp_{100kHz}	Cp_{1MHz}
GR+0N									
Lipid [% s.m]	***	***	***	***	***	***	***	***	***
Protein [% s.m]	***	**	**	0.242	0.361	0.437	0.123	0.725	**
Starch [% s.m]	***	***	**	***	***	***	***	***	***
Hardness [N]	0.762	0.939	0.649	0.722	0.757	0.711	0.973	0.742	0.699
L*	*	0.132	**	0.206	0.185	0.202	0.143	0.190	0.196
a*	0.499	0.548	0.418	0.580	0.570	0.577	0.515	0.572	0.577
b*	0.479	0.250	0.447	0.977	0.911	0.965	0.678	0.928	0.993
GR+80N									
Lipid [% s.m]	**	***	0.086	***	***	**	**	***	**
Protein [% s.m]	0.201	***	0.084	*	*	*	0.774	**	**
Starch [% s.m]	0.073	***	0.356	***	**	*	***	**	*
Hardness [N]	0.312	**	*	0.096	**	***	0.091	**	***
L*	0.263	*	0.761	*	0.071	0.191	*	0.109	0.233
a*	0.431	*	0.093	0.095	*	*	0.092	*	*
b*	*	0.586	0.099	0.584	0.767	0.756	0.666	0.787	0.780
GR+120N									
Lipid [% s.m]	0.484	***	***	***	***	**	0.627	**	***
Protein [% s.m]	***	***	***	*	*	*	0.439	**	***
Starch [% s.m]	0.642	***	***	***	**	*	0.962	**	***
Hardness [N]	0.262	0.123	0.128	*	***	***	0.239	0.137	0.122
L*	*	***	***	0.060	***	***	**	***	***
a*	0.900	0.641	0.843	0.267	*	*	0.906	0.823	0.850
b*	***	***	***	0.708	*	**	***	***	***

*** - significance level $p<0.001$; ** - significance level $p<0.01$; * - significance level $p<0.05$

all grain quality traits tested in this study.

The Principal Component Analysis enabled discriminating a sub-set of electrical parameters (additional variables) from a large group of the evaluated physicochemical parameters and identifying the usability of the analyzed electrical parameters for quality assessment of the grain of durum wheat grown in Poland. In addition, only one agro-engineering factor was considered, i.e. wheat cultivation with/without GR, to verify the versatility of the proposed electrical method – Fig 3A, and Fig 3B (with no account taken of the nitrogen fertilization factor)

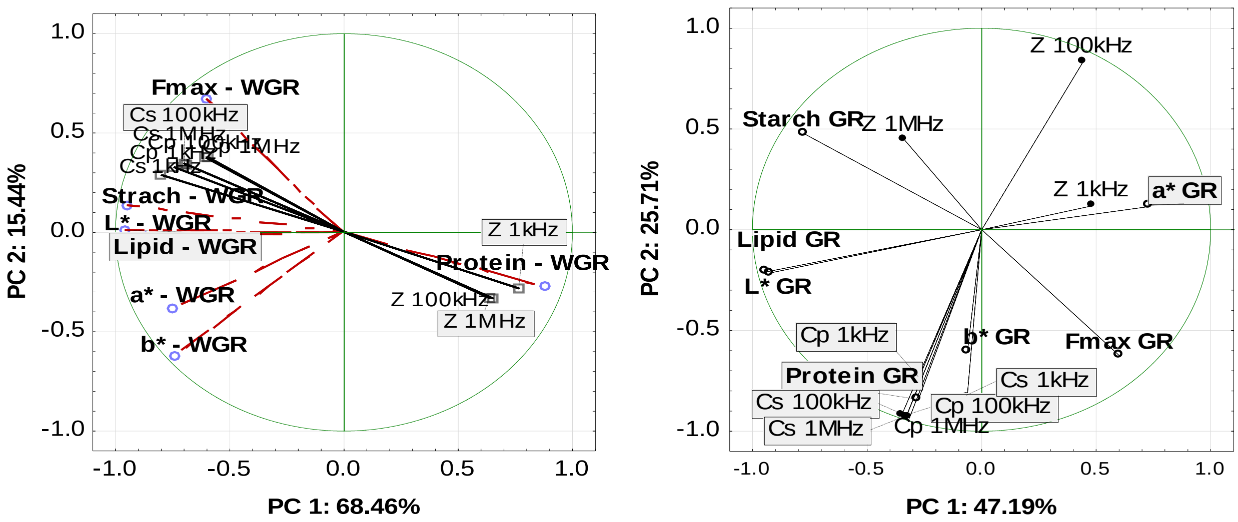

Fig. 3. Visualization of correlations between quality traits and electrical properties of the grain of durum wheat cultivated WGR (3A) and with the application of the GR (3B)

The matrix correlation between the analyzed parameters of the grain produced in the WGR and GR variants enabled discriminating two Principal Components explaining 83.90 % (Fig. 3A) and 72.90% (Fig. 3B) of the total variance of the analyzed parameters. In the first wheat cultivation variant – WGR (Fig. 3A), the visualization of the analyzed parameters (vector length, angle between vectors) onto a two-dimensional plane, defined by PC1 (68.46%) and PC2 (15.44%), demonstrated correlations between the chemical composition of the grain and its conductive (Z) and capacitive (C_p, C_s) parameters. Significant ($p<0.05$) and the strongest positive correlations were found between: protein content and $Z_{1kHz-1MHz}$ ($0.839<r<0.900$); starch content and $Cp_{1kHz-1MHz}$ ($0.807<r<0.912$) and $Cs_{1kHz-1MHz}$ ($0.874 <r< 0.946$); as well as lipid content and $Cp_{1kHz-1MHz}$ (0.624

$<r<$ 0.745) and $Cs_{1kHz\text{-}1MHz}$ ($0.700<r<0.785$). In turn, significant ($p<0.05$) and the strongest negative correlations were demonstrated between: protein content and $Cp_{1kHz\text{-}1MHz}$ ($-0.817 <r< -0.897$) and $Cs_{1kHz\text{-}1MHz}$ ($-0.867 <r< -0.916$); starch content an $Z_{1kHz\text{-}1MHz}$ ($-0.846 <r< -0.930$); as well as lipid content and $Z_{1kHz\text{-}1MHz}$ ($-0.664 <r< -0.763$). The positive correlations between protein content and Z values are most likely due to the generally higher protein content in the grain from wheat grown in the WGR variants (Tab. 3). Our previous study (Banach et al. 2017) demonstrated significant correlations between impedance and admittance values and the chemical composition (including especially lipid and starch contents) of the grain of various cereal species (wheat, barley, rye, triticale, oats) and indicated the viability of conductive parameters (measured in a frequency range of f=100 kHz-2 MHz) in discriminating cereal species and cultivars.

In the second variant of wheat cultivation – GR (Fig 3B), the visualization of the analyzed parameters was not that explicit as in the WGR variant (Fig 3A). Significant ($p<0.05$) and the strongest correlation was found only between $C_{p1kHz\text{-}1MHz}$ ($0.903 <r< 0.925$) and $C_{s1kHz\text{-}1MHz}$ ($0.902 <r< 0.937$) parameters and protein content. In contrast, significant ($p<0.05$) negative correlations were determined only between impedance value and protein content, color (b* and L*), and hardness. The values of the correlation coefficient were as follows: protein-Z_{100kHz} ($r = -0.933$); b*-Z_{1MHz} ($r = -0.801$); L*-Z_{1MHz} ($r = -0.688$); and hardness -Z_{1MHz} ($r = -0.609$). Given the above, it was concluded that the electrical method can be employed to assed the quality of durum wheat grain produced in the WGR cultivation variant. A lack of explicit correlations between the analyzed traits of grain from the GR variant points to the limited applicability of this method. The administration of the growth regulator (GR variant) had a significant effect on differences in the chemical composition of the grain, including especially contents of starch and lipids (Tab. 1), which determine grain microstructure. According to Patel et al., (2006); Nilesh et al., (2012); and Ropelewska et al., (2019), GR application elicits a positive effect on: thousand seed weight, bulking density, width of common wheat grain, grain yield, and HI, and may improve technological processes in the cereal processing industry. The grain from the GR+0N variant had a significantly lower protein content than the WGR+0N grain. Contrary to the available literature data (Łuczycka 2009; Majewska et al. 2008; Żywica 2010;Banach et al. 2017, 2020), no explicit correlations were found between the quality traits and electrical properties of the grain from wheat cultivated with GR. Presumably, the application of a growth regulator (Medax 350 S.C.) induces changes in kernel structure. Therefore, it seems advisable to identify deeper mechanisms underlaying effects of this agro-engineering factor to elucidate trends of these changes.

Summary and conclusions:

1. The cultivation of durum wheat in different variants of agro-engineering factors (GR-WGR and nitrogen fertilization: 0, 80, 120 $kg \cdot ha^{-1}$) enables modifying grain quality, including especially starch and lipid contents. The highest contents of starch and lipids, and the highest hardness of the grain can be achieved in wheat cultivation variants with or without growth regulator (GR of WGR) without nitrogen fertilization (0N). In turn, the highest protein content of the grain can be obtained upon nitrogen fertilization at 120 $kg \cdot ha^{-1}$. These results suggest the feasibility of tailored optimization of grain production in durum wheat processing.
2. The analysis of correlations between the quality traits and electrical parameters of the grain demonstrated cultivation variant-dependent differences in the strength of these correlations. In the grain from the WGR+0N variant (environmentally friendly), the strongest correlation was found between the conductive and capacitive parameters, in the entire range of current frequencies. Coupling the WGR cultivation variant with higher nitrogen fertilization rates (80 and 120 $kg \cdot ha^{-1}$) caused strong correlations ($p<0.001$), indicating the viability of capacitive parameters (C_p and C_s) measured at higher frequencies (100kHz; 1MHz) or Z_{1MHz} in grain quality assessment. GR application during wheat cultivation impedes the use of electrical parameters for grain quality evaluation; however, the feasibility of using C_s parameter at f=100kHz and f= 1MHz was strongly indicated in the GR+120N variant.

3. The agro-engineering factor of growth regulator (GR) application during native durum wheat cultivation hampers the use of the analyzed electrical method to assess its grain quality (chemical composition, hardness, and color). This type of assessment can only be made using one of the conductive parameters, i.e. impedance, measured at high frequencies (100 kHz; 1MHz). The quality of the grain produced in the WGR variant can be effectively evaluated using conductive (Z) and capacitive (C_p, C_s) parameters in the entire tested range of measuring current frequencies (1kHz-1MHz).
4. The cultivation of durum wheat in the integrated system, especially in the WGR variant, facilitates grain quality modeling and enables using a non-invasive electrical method for a rapid assessment of the quality traits of the grain, while raising no concerns over natural environment safety.

Funding:

The research was financed by the "Project financially supported by Minister of Education and Science in the range of the program entitled "Regional Initiative of Excellence" for the years 2019-2022, Project No. 10/RID/2018/19, amount of funding 12.000.000 PLN" and the University of Warmia and Mazury in Olsztyn, Poland (projects: No. 22.610.100-110; No. 17.610.012-110; No. 22.610.100.115).

Competing interests: The authors have declared that no competing interests exist.

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