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Dynamic NIR Spectroscopy to Monitor Wheat Product Processing: A Short Review

¹A. Aït Kaddour and ²B. Cuq

¹UR Typicité des Produits Alimentaire, Département QEA, VetAgroSup Campus Agronomique de Clermont, 89 Avenue de l'Europe, BP 35, F-63370 Lempdes

²UMR 1208 Ingénierie des Agropolymères et Technologies Emergentes, CIRAD, Inra, Montpellier SupAgro, Université Montpellier 2, F-34000 Montpellier

Corresponding Author: A. Aït Kaddour, UR Typicité des Produits Alimentaire, Département QEA, VetAgroSup Campus Agronomique de Clermont, 89 Avenue de l'Europe, BP 35, F-63370 Lempdes Tel: 33 (0)4 73 98 13 78 Fax: 33 (0)4 99 61 30 76

ABSTRACT

The aim of this short review is to present the different applications of NIR spectroscopy to monitor and describe physical and chemical modifications during wheat products processing. The dynamic NIR spectroscopy for cereal products concerns mostly in-line monitoring of unit operations involved in transformation of wheat flour to food products. NIR spectroscopy has been mainly investigated as an in-line method to monitor bread dough mixing. Other applications such as flour agglomeration process, pasta extrusion and lamination process, dough proofing, thermal treatments and product storage have also been investigated.

Key words: Bread, starch, proofing, agglomeration, gelatinization, retrogradation

INTRODUCTION

NIR spectroscopy is probably one of the physical methods best adapted to analyse wheat products. Analytical NIR spectroscopy is a useful and cost effective method to analyse ingredients, processes and product stages of production. It has been used for routine inspections for three decades. There are 3 main ways in which NIR spectroscopy is currently applied in wheat products. The 1st is that of straightforward analysis, for the accurate, reliable (precise) and very rapid determination of wheat grain and derived products composition. The 2nd is to act as a screening method in plant-breeding (for the selection of cross-breeds with the desired qualities) and in wheat grain diagnostics at the time of delivery. The 3rd is to use NIR spectroscopy as an in-line tool to monitor physical and chemical changes in wheat grain and derived products during processing.

The use of NIR spectroscopy as an in-line monitoring tool in cereal processing has opened a new field of applications, whereby NIR spectroscopy can provide information on changes in chemical and physico-chemical characteristics of foods during manufacturing processes (Miller, 1979; Robert *et al.*, 1999; Osborne, 1999, 2000, 2006; Bertrand and Dufour, 2000; Dowell and Maghirang, 2002; Wellner *et al.*, 2005). NIR spectroscopy method has been defined as dynamic NIR spectroscopy by Kaddour *et al.* (2007). Dynamic NIR spectroscopy provides information that is relevant to calculate the reaction rate constants for the process. Dynamic NIR spectroscopy gives complementary information with those that can be obtained using classical analytical methods, such as rheology or chemical analysis. The potential applications of dynamic NIR spectroscopy

for cereal products concern in-line monitoring of unit operations involved in transformation of wheat flour to food products (flour agglomeration, dough mixing, pasta dough extrusion and sheeting, dough proofing, thermal treatment and storage).

The aim of this short review is to present the different applications of the dynamic NIR spectroscopy as a tool to monitor processing and to describe the physical and chemical modifications during wheat processing.

NIR MONITORING OF BREAD DOUGH MIXING

Wesley *et al.* (1998, 2002) patented original approach of NIR spectroscopy technique to monitor dough mixing. The changes in NIR spectrum *versus* mixing time were considered to construct the NIR mixing curve and to determine the optimum mixing times. Several research groups in Europe (Alava *et al.*, 2000; Alava *et al.*, 2001; Millar *et al.*, 2000, 2001; Millar and Alava, 2002; Kaddour *et al.*, 2005, 2007, 2008a, b) and USA (Psotka *et al.*, 1999; Psotka and Olewnik, 2000; Huang *et al.*, 2001; Dempster *et al.*, 2004) have confirmed the ability of NIR spectroscopy to monitor dough mixing and extended applications over different mixer systems, wheat types and dough formulations. In-line measurement of chemical changes in dough by NIR spectroscopy was conducted by Alava *et al.* (2001), Wesley *et al.* (1998) and Kaddour *et al.* (2007). Those different authors used classical analysis of spectral data (variation of peak area and peak high during mixing time) and chemometric methods (principal components analysis, 2 dimensional correlation spectroscopy methods, etc.) to extract chemical information from NIR spectra. The use of NIR technology in breadmaking process due to its non invasive aspect would lead to both the industrial automation of the mixing process and the better description of physico-chemical phenomenon implied during dough mixing. A more detailed literature analysis on the NIR monitoring of bread dough mixing was presented in Kaddour and Cuq (2010) review.

APPLICATION TO MONITOR PROOFING OF YEASTED BREAD DOUGHS

Downey and Sinnelli (2005) applied NIR and MIR spectroscopy techniques to a preliminary study of changes in wheat flour doughs during the proof phase. Spectra were collected contemporaneously by NIR (750-1100 nm) and middle-IR (700-2000 cm^{-1}) using a fiber optic probe and a horizontal ATR cell, respectively. PCA was used to follow changes in the recorded spectra signals overtime. Using NIR in particular, the rate of the process accounting for the major source of spectral variability was determined to reach a maximum in strong baker's flour at about 22 min, the time period normally used for dough proofing in the Chorleywood Bread Process. When xylanase enzymes were incorporated in such doughs, the kinetic parameters of this process were seen to alter. Using both NIR and MIR data allowed molecular interpretations of the major spectral events to be made. More recently, Nicoletta *et al.* (2007) proposed NIR spectroscopy as a valid tool to on-line monitor the dough proofing using different types of flours. A FT-NIR spectrometer and a VIS/NIR monochromator were used. For both techniques a fiber optic probe positioned at the dough surface was used. PCA of second derivative NIR spectra allowed to identify on the loading spectra associated to PC2 and PC3 NIR bands relative to water and starch/proteins respectively. The analysis of the scores during proofing time shows that the time at the minimum PC2 score value was associated to the time where the dough collapses and the time at the first maximum value of the PC3 score was associated to the maximum dough development. The method was validated for different dough formulations and temperatures.

The proofing analysis using the VIS/NIR spectroscopy (750-1100 nm) demonstrated that PC1 and PC2 were correlated to the proofing time. The loading associated to PC1 was describing variation in starch and water vibrations. The first derivative of the PC1 scores trace during proofing time allowed to predict the maximum dough development time. The analysis of the starch band at 978 nm during proofing time allowed to calculate the same proofing times as those obtained after PCA of second derivative NIR spectra.

WET AGGLOMERATION OF WHEAT FLOUR DURING MIXING

In pasta and noodle manufacturing, the mixing process under low water content is a key unit operation that ensures a uniform distribution of ingredients and a production of an evenly particle size distribution for further processing (Hatcher and Preston, 2004; Huang, 1996; Kim, 1996). The partial agglomeration of wheat flour (or semolina) in presence of low water content (at 30-33%) and the formation of small agglomerates of dough is a condition to ensure good processability of the dough agglomerates obtained at the end of mixing (Oh *et al.*, 1983, 1985a-c). Changes in crumbly dough properties during mixing of wheat flour are mainly due to physical modifications of particles through agglomeration mechanisms (particle size distribution and agglomerate density) and also to some chemical modifications of wheat components (proteins aggregation levels) (Kaddour *et al.*, 2007). Empirical knowledge of operators are still the current tool to manage the wet agglomerates of wheat flour during mixing and to identify the time necessary to obtain an homogeneous water and particle size distributions. In-line probe measurements are not available to continuously monitor the formation of agglomerates during the crumbly dough mixing. Some works have been conducted in the pharmaceutical industry to investigate the ability of NIR spectroscopy to monitor and control the agglomeration rates of drugs (Rantanen and Yliruusi, 1998). Frake *et al.* (1997) demonstrated that NIR infrared spectroscopy can be used to in-line monitor granule water uptake and particle size changes during aqueous top-spray fluid bed granulation. Based on those researches Kaddour *et al.* (2007) investigated the ability of NIR spectroscopy to characterize wheat flour agglomeration during wet mixing. Actually, this study is the only one that tried to monitor and describe by NIR spectroscopy the agglomeration process observed during wheat flour mixing. A FT-NIR Analyser equipped with a fiber optic probe directly in contact with the crumbly dough allowed them to record NIR spectra. Different level of NIR spectra analysis was proposed by the authors. The first level was the direct analysis of the raw spectra. The second level was the analysis of NIR spectra after second derivative and the third one was the analysis of raw and second derivative spectra by PCA. Those different analytical levels were very interesting because they allowed them to propose a deep analysis of the NIR spectra modification. For example the increase of the raw spectra baseline was assigned to a modification in the light scattering properties of the flour during agglomeration suggesting that this level of analysis is highly associated to physical modification (size) of wheat flour agglomerated. The analysis of the second derivative spectra demonstrated absorption features at specific wavelengths (1157, 1202, 1349, 1426, 1583, 1702, 1777, 1922 and 2100 nm). Those peaks were assigned to chemical changes during agglomeration of wheat flour in presence of water (Osborne and Fearn, 1986) and more particularly to C-H vibration of the C-H₃ group (1157, 1202, 1702 and 1777 nm), to the starch vibrations (first overtone of O-H intermolecular vibration at 1583 nm and the combination of the O-H deformation and C-O stretching at 2100 nm) and to the C=O stretching second overtone of the CONH group (1922 nm). The PCA permits to construct the NIR agglomeration curves assigned to the variation of physical and chemical phenomenon observed during mixing. Mathematical modeling of the PC2

score curves allowed them to determine the turning point at which the rate of change of the trace was a maximum. This time (called the NIR agglomeration time) was found to be similar to the wet agglomeration times defined as the time allowing agglomeration mechanisms to occur and the time necessary to reach constant particle size distribution inside the mixer (Kaddour *et al.*, 2007). NIR mixing curves constructed using second derivative NIR spectra allowed them to propose a mathematical model to determine the mixing time corresponding to the time at which a constant PC₁ scores values are reached. This first study demonstrated not only the potential of NIR spectroscopy to monitor wet agglomeration of wheat flour but also the possibility of this method to describe physical and chemical modifications observed during the agglomeration process. More studies are necessary to confirm those first results by modifying the agglomerate formulation (water quantity, flour type, ingredient, mixer bowl, shape of the mixer harms). Moreover the chemical assignment of NIR spectral bands has to be confirmed for example by coupling the NIR spectra to an other spectroscopic method (MIR spectroscopy, fluorescence spectroscopy).

APPLICATION TO MONITOR FRESH PASTA PROCESSING

Zardetto and Rosa (2006) were also interested by the potential of using dynamic NIR spectroscopy to investigate the chemical differences that can be observed between two processed pasta products. They evaluated the ability of NIR spectroscopy to discriminate the dough structure of fresh pasta produced by a lamination process or by an extrusion process. The PCA approach was used to detect the difference between extruded and sheet rolled pasta. The PC1 and PC2 represented 88% of the total variability of the NIR spectra. PC2 showed higher dispersion within the extruded samples, due to the different specific mechanical energy input values during processing. PC1 is the component that better separates the two pasta types. The loading plot of PC1 is characterized mainly by 4 absorption bands at 1115, 1460, 1940 and 2229 nm. The band at 1460 nm corresponds to the first overtone of the O-H stretch vibrations. The difference in this absorption band is thought to be due to different states of the hydrogen bonding between starch and water. These slight differences in NIR absorption band associated with the hydroxyl groups that are intermolecular hydrogen bonded could be explained by higher impact of extrusion (than lamination) on the structural and molecular characteristics of the starch chains. The band at about 1940 nm can be attributed to free water (Apruzzese *et al.*, 2000). This study is very interesting since it is complementary to the study conducted by Kaddour *et al.* (2008a, b). This demonstrated that NIR spectroscopy method could be used as a tool allowing to monitor physical and chemical modification and to control the process of pasta processing.

STARCH GELATINISATION: APPLICATIONS TO MONITOR THERMAL TREATMENTS AND EXTRUSION-COOKING

Because NIR spectroscopy is applicable to the study of chemical changes involving hydrogen bonds, applications concerning the description of chemical changes in starch structure occurring during thermal processing of cereal foods have been investigated (Osborne, 2000). Accordingly, NIR has been used to study gelatinisation (and also retrogradation) processes in wheat starch systems.

Wesley and Blakeney (2001) investigated the ability of NIR spectroscopy to describe the process of starch gelatinisation induced by controlled thermal treatment using a modified Rapid Visco Analyser (RVA) system. Different starch, protein and water mixtures (as samples are heated and sheared) were characterized during RVA gelatinization. Dynamic approach of NIR spectroscopy was obtained by direct analysis of the changes in 2nd derivative NIR spectra measured as a function

of time. Major absorbance changes were found at 1160 and 1400 nm that may be associated with chemical changes as heating progresses. Because some of the variation in NIR spectral intensities could be due to changes in the sample temperature, they determined the relative effects of chemical changes and temperature on the optical properties during the transition from a particulate suspension to a clear gel, a series of spectra using starch-titanium dioxide (unreactive fine particulate solid that disperse easily in water) mixture was recorded. If enough titanium dioxide is present, then the scattering due to the titanium dioxide will mask a change in the light scattering coefficient of the starch water mixture. The only feature of note on NIR spectrum data recorded using titanium dioxide is the broad hump in the profile of the water absorbance peaks at 1160 and 1400 nm, as the temperature of the sample rises and falls. Thus, any dramatic changes in the NIR spectrum that occur during the experiment are unlikely to be due to effects of temperature on the NIR spectrum. Wesley and Blakeney (2001) also indicated that the spectral changes that occur when starch is gelatinized are not due to changes in the scattering properties of the sample. Wesley and Blakeney (2001) postulated that the absorbance feature of interest is at 1160 nm. The change in the 1400 nm region of the spectrum consists of a combination of an absorbance change and a wavelength shift. The change at 1160 nm was assigned to the stretch-band combination mode of water and associated with the bonding of water molecules. The decrease in the absorbance can thus be interpreted as a decrease in the free water. The absorbance change at 1160 nm is shown to be associated with the gelatinization of the starch granules, a process that occurs before pasting and gelation. Initially, water is bound by the starch and gluten, although the presence of proteins decreases the amount of water bound compared to the starch-only system. The changes in the absorbance at 1160 nm also provide information on the interactions that occur when protein-starch-water mixture are heated. The results obtained suggest that in excess of water conditions, there is a complex interaction between gliadin, glutenin, starch and water which involves the initial ordering of water, increased ordering on stirring and the subsequent disordering of the water on heating, related to the presence of smaller protein molecules, occurring simultaneously with starch gelatinization.

Application to monitor thermal treatment of pasta: Zardetto (2005) investigated the ability of NIR spectroscopy (between 1000 and 2500 nm) to identify the chemical changes of fresh pasta induced by thermal treatment (steaming at 88-98°C during 2-6 min). A linear regression model ($R = 0.88$ and $SEP = 0.18$) was obtained using PLS regression for the description of thermal treatment intensity (time-temperature effect) and NIR spectroscopy data. Analysis of PLS loading spectra for the 4 factor models indicated that only three spectral regions made positive contributions to the regression equation. Factor 1 presents a loading with major positive correlations at 1760, 1730 and 1700 nm associated with the absorbance of C-H groups in wheat proteins. Other contributions to the development of this factor were wavelengths related to O-H groups in starch (2276 nm). Factor 2 had similar absorption bands to those displayed by the first loading as well as one additional band (2258 nm) which has been described as O-H/bend/hydrogen-bonded O-H stretch and another peak at 2206 nm. The third and fourth PLS loading presenting the same absorption bands to those displayed by the first and second loading as well as one additional band at 1678, 1740, 1850 and 2206 nm. Comparison of NIR spectra at different thermal treatment intensity revealed that an increase of the thermal treatment intensity is accompanied by an increase in the 1675, 1740 and 1853 nm bands and by a decrease in the 1727 and 2238 nm bands. The heat treatment produced complex changes in fresh pasta in which gelatinization of starch and the denaturation of proteins

were competing. The positive peak at 1740 nm was assigned to the SH groups because heat treatment could cause conformational changes with the formation of new intermolecular disulphide bonds but this assignment has to be confirmed by other studies. The peak at 2238 nm could be related to the spectrum of starch and provides correlative information between the spectral data and the degree of gelatinized starch. The spectrum of a thermally treated sample was compared with a spectrum of non pasteurized pasta. The thermal treatment induced an increase in intensity of the 1727 and 1810 nm bands and a decrease of the 1675, 1695, 1780, 1853 and 2238 nm bands. The most important feature was a band at 1765 nm, which was not present in the thermally treated pasta. The results indicated that most thermal processing effects involve the CH₂ and CH groups. The effect of thermal processing is to change the spectra due to the CH₂ groups in starch (1715 nm), CH groups in cellulose (1780 nm), CH₂ groups in gluten (1765 nm) and OH stretching in cellulose (1810 nm). The absorption band observed at 1675 and 1695 nm characterizes the C-H stretch. The study of Zardetto (2005) closes the process of pasta chain production initially conducted by Zardetto and Rosa (2006) and Kaddour *et al.* (2007). This implies that NIR spectroscopy is an interesting tool that can be used not only to control the chain of pasta production but also to understand and monitor physical and chemical modification occurring during this process.

Application to monitor extrusion-cooking: Extrusion cooking is used to manufacture a wide range of products from cereals including breakfast cereals, snacks foods and pet foods (Osborne, 2000). Conventionally, control is attempted by measurements of inputs such as moisture content, screw speed, barrel temperature, etc. In contrast to breadmaking where the product structure is largely controlled by protein development, starch is the chief functional ingredient in extrusion-cooking. The extrusion cooking of cereal-based raw materials can involve the melting of the starch granules followed by dispersion into a continuous phase and partial depolymerisation. The extent of transformation of the raw material, referred to as the degree of cook, is crucial to final product quality. Degree of cook increases when there is an increase in depolymerisation of the starch molecules which in turn results in an increase in the number of free hydroxyl (O-H) bonds and a decrease in paste viscosity. NIR spectroscopy was tested to monitor changes in starch structure during extrusion-cooking.

Out-line monitoring: Guy *et al.* (1996) have shown that NIR reflectance spectroscopy could be used to determine the extent of processing of dried powdered extrudates, prepared from wheat starch, white and whole-meal flours. The wavelength region of interest for degree of cook measurement was found at 1400-1500 nm. The spectral patterns associated with the description of extent of processing show that overall absorption at 1460 nm increases while absorptions at 1528 and 1580 nm both decrease with increasing the extend of processing. This implies that NIR measurements describe a progressive cleavage of hydrogen bonds between hydroxyl groups within the starch molecules with increasing severity of processing (Guy *et al.*, 1996). There was a causal relationship between the intensity of the free O-H band in starch at 1430 nm and the extent of depolymerisation of the starch (Millar *et al.*, 1996). Osborne (1996) identified 4 key wavelengths around 1410, 1430, 1460 and 1520 nm that are associated to starch gelatinization in extrusion cooking systems. These wavelengths are within the O-H stretch first overtone region and may be assigned to different states of hydrogen bonding of OH in water and starch. Absorptions at 1412, 1466 and 1510 nm were assigned to OH in water with no hydrogen bond (W0), with one hydrogen bond (W1) and with two hydrogen bonds (W2). Absorptions at 1430 and 1520 nm may be assigned

to OH in starch with no hydrogen bond (S0) and with one hydrogen bond (S1). The hydrogen bonding of the OH in starch could be intramolecular or intermolecular to water molecules. For starch gelatinization, the S0 decreases, while the W0 and W1 increase.

On-line monitoring: Since successful calibrations were obtained using the powdered samples, the measurement was attempted on-line. The spectral characteristics of the different forms of starch found in the hot melts close to the die of the extruder were identical to those of the powdered extrudates. Millar *et al.* (1996) used a fiber optic reflectance probe that was installed in the die of the extruder and the light was transmitted through the melt and reflected back from a polished steel pillar. Because the extrudate melts are glassy and do not back-scatter NIR radiation, Evans *et al.* (1999) used NIR transmittance probes located inside a special die block. The residual absorbance was calculated from absorbance at 1426 nm and from peak area between 1400 and 1500 nm. Analysis of cross-correlation demonstrated that NIR measurements on extrudate melts correlated well (0.85-0.89) with the measurements of the degree of cook made on powdered extrudates. NIR measurements were associated to the increase in free O-H as the starch is progressively depolymerized.

STARCH RETROGRADATION: APPLICATION TO MONITOR BREAD STALING

Starch retrogradation is a complex process that occurs during storage of most of the starchy foods. During storage of starchy systems after gelatinization, both the amylose and amylopectin molecules rearrange and the rigidity increases. Starch retrogradation is accompanied by an increase in crystallinity of structure, as H-bonds between starch molecules re-form (Miles *et al.*, 1985). The retrogradation process may proceed in two different stages, including short-term gelation of amylose within the continuous phase, which occurs as the first step of retrogradation completing within a few hours and subsequent long-term crystallization of amylopectin, which occurs at a much slower rate (requiring several weeks).

Different studies have demonstrated that bread staling and starch retrogradation mechanisms could be followed by NIR spectroscopy (Wilson *et al.*, 1991; Osborne, 1996, 1998). Wilson *et al.* (1991) first studied white bread staling using NIR spectroscopy. Rate constants were calculated directly from plots of second derivative NIR data *versus* time. The staling rate measured by NIR spectroscopy agreed with that obtained from DSC measurements. Xie *et al.* (2003) found strong correlations ($R^2 = 0.68-0.90$) between NIR measurements and firmness changes occurring during bread staling. Xie *et al.* (2004) investigated the potential of NIR spectroscopy as a fundamental tool to study bread staling, with the help of DSC. NIR spectra correlated strongly ($R^2 = 0.91$) with DSC measured amylopectin retrogradation data. On the other hand, they demonstrated that NIR spectroscopy correlate poorly ($R^2 = 0.21-0.40$) with the 1 amylose-lipid complex retrogradation data measured by DSC. NIR spectroscopy had difficulty in measuring the retrograded amylose-lipid complex, since amylose-lipid complex did not change as much as the amylopectin did while in storage. Three important wavelengths (970, 1155 and 1395 nm) were identified in the beta coefficients obtained when 3 factors were used for the correlation with DSC measured amylopectin retrogradation data. The peak at 970 nm might correspond to the second overtone of O-H and might be due to moisture and starch structure changes. The peak at 1155 nm might be due to the second overtone of C-H3 and the peak at 1395 nm might relate to the second overtone of C-H, C-H2 and C-H3. These peaks might correspond to starch structure changes (Xie *et al.*, 2004).

NIR spectroscopy not only follows physical changes in bread staling, but also provides chemical information. Three changes could be described by NIR spectroscopy during bread staling: crumb scattering, moisture loss and starch structure changes. (1) Crumb scattering refers to the light-scattering properties of the crumb. Because NIR detects kernel texture by virtue of its effect on scattering, it is reasonable for NIR spectroscopy to follow bread staling in an analogous manner because starch crystallinity changes crumb physical properties, such as scattering of NIR radiation. (2) Moisture loss. It was reported that the bread staling rate constant calculated from NIR spectroscopy absorbance at wavelength 1934 nm was in close agreement with that obtained by DSC. After scattering correction, absorbance at 1934 nm had no correlation with storage time, but a high correlation with moisture content (Wilson *et al.*, 1991; Osborne, 1998). (3) Starch structure changes due to inter- and intra-molecule hydrogen bonding. Starch crystallinity is extensively hydrogen bonded, both intramolecularly and intermolecularly to water. Absorbance generally decreased over the whole range as crystallinity developed during bread staling. This may be due to starch crystallinity development in bread (Wilson *et al.*, 1991; Osborne, 1998). It was observed that the rate constants for retrogradation process in bread calculated from the second derivatives of $\log(1/R)$ at wavelengths 1414 (or 1412) and 1465 (or 1466) nm were similar to those obtained from DSC measurements (Wilson *et al.*, 1991; Osborne, 1998). Those wavelength bands were previously assigned to different hydrogen-bond states of water in food such as intra- and intermolecular hydrogen bonds. Osborne (1996) stated that spectra other the range 1350-1650 nm provide information about changes in starch structure during bread staling. For bread retrogradation it was observed that the W0 decreases, while the W1 and W2/S1 increase. This is consistent with the hypothesis that bread retrogradation, is accompanied by an increase in crystallinity in the starch fraction of the crumb as hydrogen bonds between starch and water reform.

CONCLUSIONS

NIR spectroscopy demonstrated its capability to in-line monitor different wheat processing. This method is well adapted to identify and describe physical, chemical and physicochemical modifications occurring during wheat processing. The different literature data demonstrated that the NIR spectroscopy can be used to generate relevant information to monitor wheat processing because it is the least perturbing method of exploring molecular changes, during the input of mechanical energy. It can be considered as a valuable method for the industry to analyze, control and understand the origin and construction of wheat based products quality. Nonetheless, NIR spectroscopy is a physical method sensitive to overtone and combination of fundamental vibrations that makes difficult the assignment of NIR absorption bands to a specific molecular vibration. An alternative way to solve this problem efficiently is to combine different detection techniques with NIR spectroscopy, such as fluorescence spectroscopy, MIR spectroscopy and UV light spectroscopy.

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