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# Development of Rapid Analytical Methods for Determining Corn Quality

by

**Anuchita Chawnua**

**A DISSERTATION**

Presented to the Faculty of

The Graduate College at the University of Nebraska

In Partial Fulfillment of Requirements

For the Degree of Doctor of Philosophy

Major: Food Science and Technology

Under the Supervision of Professor Randy L. Wehling

Lincoln, Nebraska

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GRADUATE COLLEGE
UNIVERSITY OF NEBRASKA

# Development of Rapid Analytical Methods for Determining Corn Quality

Anuchita Chawnua, Ph.D.

University of Nebraska, 2000

Advisor: Randy L. Wehling

The study proposed to develop a rapid method for classifying corn according to drying temperature, to develop methods for predicting corn quality characteristics relating to wet-milling, dry-milling and alkaline cooking, and to improve a high performance liquid chromatography (HPLC) technique for measuring fumonisin $B_1$ ($FB_1$) in extruded corn.

To evaluate the ability of Near Infrared Spectroscopy (NIRS) to classify dried corn according to drying temperature, whole kernel corn samples were dried under different temperatures. Spectra were collected from the visible and near infrared regions. Discriminant analysis based on Mahalanobis distances was applied to classify the samples. The results indicated that NIRS was a promising technique for classifying corn by drying temperature, as correct classification rates of 84.3% were achieved.

Corn characteristics, including starch yield, tangential abrasive dehulling device (TADD) index, dry matter loss (DML), and nixtamal moisture content, were measured using NIRS. In establishing calibration models, Partial Least Squares (PLS) and Multiple Linear Regression (MLR) were applied. The results revealed that NIRS has the ability to predict starch yield with a high correlation coefficient of validation (r-value = 0.898).

A high drying temperature lowered the ability of NIRS to predict. For TADD index evaluation, the study suggested that NIRS has potential to predict this parameter, and drying temperature had no affect on its ability. In the assessment of the ability of NIRS to predict corn characteristics for alkaline cooking, the study showed that the ability of NIRS to predict DML was poor, but the technique could successfully predict nixtamal moisture content. The drying temperature had a slight effect on the NIRS prediction.

Use of an enzyme to extract $FB_1$ from extruded corn prior to HPLC determination and the use of 6-aminoquinolyl-n-hydroxysuccinimidyl carbamate (AQC) for derivatizing $FB_1$ were investigated. The results indicated that using an amylase enzyme significantly improved $FB_1$ recovery. The study also showed that AQC was a good derivatizing reagent for $FB_1$, as it gave a more stable fluorescent compound than the o-phthalaldehyde derivative now commonly in use.

## Acknowledgements

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# PART I OVERVIEW

Corn is the largest agricultural commodity produced in the United States. Although the amount of corn used for food and industrial products is relatively small compared with the annual level employed for animal feed, the amount of corn used for food and industrial products is a significant portion of the corn economy and has been increasing every year. The major industries are wet milling, dry milling, fuel alcohol, and other related industries. Corn sold through commercial channels is based on some standard qualities, which generally, provide only basic information to allow buyers and sellers to agree on a price. Often times corn purchased based on standard grades gives unsatisfactory yields when it is used for processing into a particular product. Corn quality is affected by many factors; one of most critical factors is the drying process after corn is harvested. Drying corn too rapidly at excessive temperatures lowers corn quality with respect to its commercial use. Even though several tests have been established to screen for corn that is suitable for certain end-uses, those methods are time consuming, labor intensive, and the agreement between laboratory results and industrial outcomes are often poor. It is essential to define corn quality criteria for specific intended uses. In addition, rapid methods to evaluate the end use attributes of raw corn are needed.

Recently, the popularity of near infrared (NIR) spectroscopic analysis has steadily increased due to its ability to quickly provide analyses on diverse materials, in both qualitative and quantitative analyses. The NIR technique has proven to have the ability to discriminate among a number of materials for quality control. In quantitative analysis, several studies have been published showing that the NIR technique is feasible and reliable to predict several quality factors of various grains including corn, but no

study has investigated the effect of drying temperature on the ability of NIR to determine those quality aspects. Therefore, this study was carried out to investigate the potential of NIR to classify corn dried at different temperatures. Moreover, the present study was conducted to evaluate the ability of NIR spectroscopy to predict quality characteristics of corn including starch yield from wet milling, hard endosperm as a measure of dry milling yield, and dry matter loss and moisture content of nixtamal from alkaline cooking, as well as the effect of drying temperature on the ability of NIR to predict these traits.

Apart from studying NIR spectroscopic applications, this study was also carried out to improve methodologies for determining corn quality in the area of mycotoxin contamination. Mycotoxin contamination of corn is a serious quality problem and difficult to regulate. There are several groups of mycotoxins. Fumonisin is one group of the most concern. Up to this time, only a few studies have been done to develop analytical methods for quantitation of this toxin. New analytical technologies are greatly needed to improve accuracy and sensitivity of fumonisin analysis.

This dissertation is made up of four parts including, part I, overview, which provides the general contents of this paper; part II is a literature review containing three sub topics, corn production and industry, NIR spectroscopy, and mycotoxins with fumonisin highlighted. Part III describes the research topics in six chapters; (1) Classification of Artificially Dried Corn by Drying Temperature Using Near Infrared Spectroscopy (NIRS), (2) Effect of Drying Temperatures on Starch Yield Determination by NIRS, (3) Influence of Drying Temperatures on NIRS Prediction of Hard Endosperm of Whole Kernel Corn, (4) Determination of Alkaline Cooking Characteristics of Corn

Using Near Infrared Reflectance Spectroscopy, (5) Application of Enzyme Extraction for Fumonisin $B_1$ in Extruded Corn Grits, and (6) The Evaluation of 6-Aminoquinolyl-N-Hydroxysuccinimidyl Carbamate (AQC) as a Derivatizing Reagent for Fumonisin $B_1$ Determination. All of them detail, illustrate, and discuss the research results. The final part (part IV) represents the overall conclusions of this paper.

# PART II LITERATURE REVIEW

## 1. Corn Production and Industry

### 1.1 Corn Production

Corn has the second largest production in the world after wheat (USDA, 1999) and is the third most important cereal grain in the world, after wheat and rice (FAO, 1992). Basically, it is used for human foods, animal feeds, and serves as a basic raw material for a variety of products such as starch, oil, alcoholic beverages, corn sweeteners, fuel, and industrial chemicals. World corn production in 1997/98 was 575,334 million metric tons. Among corn producing nations, U.S. corn accounted for approximately 40 % of the world's production. Corn is the largest agricultural crop produced in this country with more acres and a higher value than any other commodity. Approximately, 9.76 billion bushels of corn were harvested in 1997/98 (USDA, 1999).



### 1. 2 Corn Quality

Corn traded through standard markets, generally is bought based on some standards. For example, in the U.S., corn is typically purchased using the U.S. Grain Grading Standards. A uniform system of grading is necessary for marketing corn to describe and provide the foundation for pricing. However, these standards provide only basic information for trading corn, and they are useful as general indicators of grain quality in market transactions. Although standards guarantee a certain level of wholesomeness, they do not ensure that corn is ideal for industrial products such as wet-milling products nor even for feeds.

In addition, some tests on raw corn may be required by government regulations, especially where the public health is concerned. An example is testing for mycotoxin contamination and chemical additives.

According to Loewer et al (1994), the properties of corn that determine the overall quality are low percentages of foreign material, broken, heat damaged and shrunken kernels, low breakage susceptibility, high milling quality, high protein content, high nutritional value, high viability, low mold content, low carcinogen content and low insect damage. However, not all characteristics are important for all applications. It depends on the intended end-use and other factors. For many applications, three to five factors are usually of interest, such as those desired for export of corn which are low moisture content, high test-weight, low breakage susceptibility and minimal stress cracks. Corn quality factors are influenced by several factors such as the climate conditions under which the corn was grown, hybrid, pre and post harvest handling, etc.



## 1.3 Effect of Drying on Corn Quality

Due to a relatively short harvest period, corn is often harvested at high moisture contents of 22-30% (Wall et al 1975), thus, rapid drying is needed to reduce the moisture in the crop to give an equilibrium relative humidity level at which mold growth rates are zero, or so low that during the anticipated storage period, spoilage is negligible. A drying system should be capable of drying high moisture grain to safe storage moisture contents without a significant reduction in grain quality. However, the drying step is still a critical operation in grain processing. Corn may be over-heated during drying and cause grain deterioration through a series of processes. Overheating occurs by allowing kernel

temperatures to reach 180°F (82°C) (Holaday 1964). A shelled corn dried in air at this temperature or higher was damaged with respect to its commercial use. Increasing drying temperature results in increased breakage susceptibility, decreased kernel density, and lowered dry-milling quality. The kernel breakage susceptibility correlated statistically with test weight, percentage of floating kernels and percentage of stress-cracked kernels. (Peplinski et al 1975, Kirleis and Stroshine, 1990).

The importance of stress crack determination is that corn lots with high stress crack levels are highly susceptible to breakage during handling. Furthermore, if the corn is intended for use in dry-milling, a high percentage of multiple cracks will decrease the yield of large flaking grits (Eckhoff and Paulsen 1996). If the corn is used for wet-milling, high stress crack levels provide lower yield since the corn may have been heated sufficiently during drying to cause protein denaturation, which in turn makes separation of protein from starch more difficult. Also, stress cracked-corn can break during handling. A high stress crack level can reduce starch recovery by 2-3%, due to loss of solids to steep water, and reduces wet-milling process efficiencies (Eckhoff and Paulsen 1996). Peplinski et al (1975) suggested that the relative humidity of air used in drying the corn must be kept at or above 60% to minimize stress crack formation in artificially dried corn.



## 1.4 Corn Industry and End-Use Characteristics

In the past, U.S corn has been grown mainly for animal feed, whereas nowadays there is increasing use of corn for food and industrial uses. The wet-milling industry is the most important area and currently accounts for 79 % of industrial use of corn (Corn