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Precocious rapeseed spoilage detection

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Abstract. Carbon dioxide (CO₂) level measurement is widely used for informative early spoilage detection in various grains. This study was intended for research of CO₂ level measurement suitability for early spoilage detection in rapeseed. Experiments were performed using grain model systems in the controlled environment. Grain model systems were prepared with rapeseed of different moisture contents (9; 11; 13; 15 %) and various temperatures (25, 30 and 35 ^oC). Research was based on previous study of CO₂ sensor suitability for wheat spoilage detection and our results showed that CO₂ sensor could not be a reliable indicator of early spoilage detecting in stored rapeseed in contrast to wheat research case.

1. Introduction

It is important to ensure good rapeseed quality until they reach consumers. The fungi is the main agent of spoilage in rapeseed because of high moisture content (MC). MC is an important factor in grain quality deterioration, as it limits the development of bacteria, fungi, mites and insects that cause spoilage in stored grains. MC and temperature are two most important factors that cause rapeseed deterioration during storage [1-4].

Detection of fungal infection at the early stage is one of the factors that can protect rapeseed from decrease of seed germination, oil quality and from increase of free fatty acids content in seeds.

Traditional methods used for spoilage detection such as chromatography, mass spectrometry or microscopic methods are long time consuming and need highly trained staff or sophisticated instrumentation.

Our previous study confirmed that CO_2 respiration rate (RR) monitoring using gas sensors could be used as an early wheat spoilage detection technique [5]. The goal of this paper was to apply the CO_2 level measurement technique for possible detection of biological contamination in initial rapesed spoilage stages instead of spoilage detection by the traditional methods such as visual inspection, smell and temperature measurement.

2. Materials and methods

2.1. Preparation of Grain Samples

The research was carried out on rapeseed that was purchased from one supplier. The initial rapeseed MC was 4.9 %. During the preparation of the experimental samples, a required rapeseed moisture content (MC) was achieved by adding certain amount of distilled water. MC values were determined from the average of three replicate measurements by applying the oven drying technique. Microbial activity in the rapeseed samples was evaluated using CO_2 RR. RR value allows comparing different experiment results. RR evaluates the grain mass, the container volume, and the CO_2 concentration. Rapeseed RR is expressed as mg of CO_2 produced per kg of rapeseed per hour, and was calculated by the following equation [6]:

$$RR = \frac{\Delta c_{CO_2} \times M_{CO_2} \times V_h}{V_m \times m \times \Delta t}, \qquad (1)$$

where Δc_{CO2} is the change of CO₂ volumetric concentration in ppm (10⁻⁶ L/L), M_{CO2} is the molecular weight of CO₂ gas 44.01 (g/mol), V_h is the volume of the headspace in the container (L), V_m is the molar volume of gas (L/mol), *m* is the mass of the rapeseed sample (kg), Δt is the duration (h) for Δc_{CO2} .

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2.2. Measuring system and experiments

Rapeseed samples were placed in the sealed 1600 mL containers with the measuring modules (Figure 1). The containers were stored in the environmental chamber with a controlled temperature ((25; 30; 35) \pm 1 °C) during the experiments. Each hermetic closed vessel contained 200 g rapeseed sample of specific MC (9; 11; 13; 15 %). The whole measurement system consisted of nine containers.

Each hermetic vessel has installed measurement module (CO₂ Engine K33 LP T/RH, SensAir AB, Delsbo, Sweden) which is able to measure CO₂ concentration, relative humidity (RH) and temperature (T).



Figure 1. The hermetic container filled with the rapeseed sample and the measurement module.

The measurement range of the CO₂ concentration sensors was 5000 ppm (parts per million volumetric) (accuracy ± 30 ppm $\pm 3\%$ of reading) and up to 10000 ppm in the extended measurement range (not specified accuracy). Sensors response time was 30 s. Temperature and relative humidity measurement range of the sensors were from 0 to 50 °C (accuracy $\pm 0,4$ °C) and from 0 to 80 % (accuracy $\pm 3\%$) up to 100 % RH in the extended range (non-condensing environment), respectively.

All measurement modules, used in the measuring system, have unique addresses and are connected to common data bus (Figure 2). PC with appropriate software was used to read the data from each module and store this data for the further analysis.



Figure 2. The structure of CO2, temperature and RH measuring system.

Using measuring system, which has been previously described, the experiments were carried out by measuring CO₂ amount in prepared rapeseed samples. Table 1 shows MC measurement data of one experiment of all samples at 35 ± 0.5 °C temperature.

Table 1. MC measurement data.

Module No.	1	2	3	4	5	6	7	8	9
MC, %	4,9	9,7	9,4	10,6	10,4	13,1	12,3	14,8	14,3

Figure 3 and Figure 4 are given as an example and present the raw data (CO_2 concentration and RH) during this experiment.

According to the Figure 3, the grain, with higher MC (modules 8, 9), emits higher amount of CO₂. During experiments, CO₂ sensors with higher rapeseed MC saturate after a few hours. In the experiments with a higher

MC in grains, CO_2 RR may be so high, that after a few hours the CO_2 level measurement sensors become saturated (CO_2 concentration 10.000 ppm) and then further analysis of the data is no longer possible. That is why the data only from the first hours after aeration of the samples are used for RR calculation.

RH at the beginning of the experiment is distributed throughout the volume of the container. During this period (approximately 300 min) RH level stabilizes, and remains the same for the rest of the experiment (Figure 4).



Figure 3. The raw data of CO² concentration measurements during one experiment.



Figure 4. Measured RH values during one experiment.

The RH value drops almost to environment RH value during the aeration process and reaches the previous RH value (Figure 4) when the container is closed after the aeration.

3. Results

The experiments were carried out by measuring the amount of produced CO_2 in rapeseed samples at different MC (Figure 5). CO_2 RR was calculated from the raw CO_2 concentration measurement results according to the equation (1) within the first 3 hours. Two different zones were separated and marked in the RR results versus time data (see Figure 5): blue zone – no visible changes of rapeseed samples; red zone – visible mold formations.

Differences highlighted in the respiration rates of rapeseed at different moisture contents. Rapeseed respiration increased as MC and/or temperature increased. The minimum RR was fixed in the rapeseed sample with the lowest MC (9 %).



Figure 5. Respiration rates of rapeseed at different moisture content levels and temperatures: a) MC 9 %; b) MC 11 %; c) 13 %; d) 15 %.

Analogue experiments with wheat grains showed that CO_2 RR was always higher in the contaminated grain samples than in the uncontaminated grain. These results were presented in the research [5]. In contrast, fungi appearance in rapeseed do not correlate with CO_2 RR growing. Also in contrast to wheat research case, higher MC do not have any evident impact to rapeseed CO_2 RR. This value remains stable or even marginally reduces irrespective of the grain are with or without visible mould (Figure 5 d).

4. Conclusions

Performed experiments with rapeseed samples at different moisture content and temperature, while simulating storage conditions, confirmed that analysis of CO₂ respiration rate could not provide objective information about storage conditions. Research results show that single CO₂ respiration rate monitoring using gas sensor cannot be a reliable indicator for initial spoilage stage detection of stored rapeseed.

5. References

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