



Device for determination of internal pressure in beer cans by measuring the force-displacement curves

Petr Gabriel^{1*}, Roman Beneš²

¹ Faculty of Mathematics and Physics, Charles University in Prague, Ke Karlovu 3, 121 16 Prague, Czech Republic

² Research Institute of Brewing and Malting, Lípová 511/15, 120 00 Prague, Czech Republic

*corresponding author: petr.gabriel@matfyz.cuni.cz

Abstract

Microbiological contamination of beer is usually manifested by the production of CO₂ and, hence, an increase in its concentration. The increased concentration of CO₂ in a closed package leads to an increase of internal pressure, which can cause a destruction of the package and have dangerous consequences. A device for non-destructive monitoring of the internal pressure in beer cans based on measurement of force-displacement curve has been developed and characterized in this study.

Keywords: beer; can; microbial contamination; force-displacement curve; measurement device

1 Introduction

Microbiological contamination of beer poses both a health risk to the consumers and also a frequent cause of deterioration of its sensory properties. One popular and commonly used packaging is cans, however they present a challenge to detection of microbial contamination. In contrast to transparent packages, optical measurement to detect the microbiological contamination without opening the sample is not possible. Microbiological contamination of beer is manifested in most cases by production of CO₂ or another gas, e.g., H₂S by the genus *Pectinatus* (Matoulkova and Kubizniakova, 2014) and its increased concentration in the packaging (Štulíková et al., 2021). An increase in the CO₂ concentration is associated with pressure increase according to Henry's law (Henry, 1803) which can cause a destruction of the package (Meier-Dörnberg et al., 2017). The destruction of the package can be dangerous for customers.

Pressure increase in beer causes an increase of internal pressure on the wall of the can. When external pressure is applied to the wall of the can, its displacement occurs, the size of which depends not only on the properties of the wall but also on the internal pressure on the wall. By measuring the dependence of the displacement of the can wall on the

developed force (force-displacement curve) can give us information about the internal pressure.

It has been demonstrated (Gabriel et al., 2023) that the measurement of the force-displacement is a suitable method to determine the internal pressure in cans. Based on this knowledge, an optimized device for measuring force-displacement curves on beer cans has been developed. The device has been described and its suitability and/or limitations for the application in brewing industry are discussed in this paper.

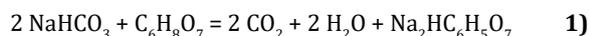
2 Materials and methods

2.1 Materials

Sodium bicarbonate NaHCO₃ (bioreagent) and citric acid monohydrate for the analysis (99%.) were purchased from Sigma-Aldrich (Germany), deionized water was prepared using Aqual 35 device (Aqual, s.r.o., Czech Republic) with conductivity under 0.2 μS/cm. Disposable 500-mL wide-mouth (full aperture) silver aluminium cans (type B64) were purchased from Kegland (Austria) (Kegland, 2023).

2.2 Solution with defined CO₂ concentration

Solution with defined CO₂ concentration was prepared according to the following reaction



Molecular weight of sodium bicarbonate is 84 g/mol, molecular weight of CO₂ is 44 g/mol, molecular weight of citric acid is 192 g/mol. One molecule of sodium bicarbonate creates one molecule of CO₂ after the release of sodium and water. The amount of CO₂ created is determined by the amount of sodium bicarbonate used, citric acid must be in excess. It is obvious that to create 1 g of CO₂, it is necessary to use 1.91 g of sodium bicarbonate, which reacts with less than 2.18 g of citric acid. Citric acid is a tribasic acid which can bind up to 3 sodium atoms depending on the pH of the solution. Equation 1 describes the reaction when citric acid binds only two sodium atoms. pK value of the reaction is much higher than pH value of prepared citric acid solution what ensures a sufficient amount of citric acid. To have an excess of citric acid during the reaction, we prepared a citric acid solution with a concentration of 21.8 g/L or all samples. This solution contains enough acid to generate CO₂ up to the concentration of 10 g/L CO₂.

2.3 Preparation of beer cans

with a defined CO₂ concentration

Cannular Compact semi-auto canning machine KL15769 was used for sample cans preparation. Aluminium cans (no. KL05449, 500ml/16.9 Oz Standard 202 end diameter with B64 lid) intended for use on KL15769 (Kegland, 2023) were used to create the samples. The cans were filled with 500 mL of citric acid solution with a concentration of 21.8 g/L. A defined amount of sodium bicarbonate was weighed to the plastic weighing bowl. The bowl was placed on the surface of the solution in the can so that the citric acid did not come into contact with the sodium bicarbonate. The can was then closed with a cap in the Cannular Compact semi-auto canning machine KL15769. After closing the can, the can was turned upside down, sodium bicarbonate came into contact with citric acid and the required amount of CO₂ was produced.

0.95 g of sodium bicarbonate was weighed into 500 mL solution to obtain a final concentration of 1.00 g/L CO₂ in the can. This corresponds to the amount of 1.91 g of sodium bicarbonate per 1 litre of solution. For higher CO₂ concentrations, the corresponding amount of sodium bicarbonate was used. Cans with CO₂ concentrations from 0 to 7 g/L with a step of 1 g/L CO₂ were prepared. Three cans of each concentration have been prepared. The cans were separated into three groups, referred as Set 1, Set 2, and Set 3. Each Set consists of all concentrations between 0 and 7 g/L CO₂.

2.4 Measurement in a commercial can

Since the Cannular – can closing machine can work only with one type of aluminium cans, lager beer Pilsner Urquell in a 0.5-L aluminium can (Plzeňský Prazdroj, a.s.) has been measured and compared to results on prepared aluminium cans with a defined CO₂ concentration.

2.5 Measurement of CO₂ concentration in used cans

After measuring the force-displacement curves, the CO₂ concentration in the cans was measured on the Carbo QC device from Anton Paar Austria according to the MEBAK 2.26.1.5 methodology (MEBAK, 2023). The measurement based on Carbo QC is contact and it is associated with opening and deterioration of the sample. The CO₂ concentration in the samples was verified with an accuracy of better than 1%.

2.6 Device for the measurement of force-displacement curves

The prototype of the device for measuring force-displacement curves on beer cans was developed at the Research Institute of Brewing and Malting in Prague, in cooperation with Faculty of Mathematics and Physics of Charles University in Prague.

The developed device uses a micrometric screw to press a punch against the can wall and a load cell (IFFL-CHA-10KG from Interface force e.V., Tegernsee, Germany) to measure the resulting force. The can is placed into the device in a horizontal position (the axis of the can is horizontal) on a plastic holder with a cylindrical surface long entire length (see Figure 1). The can holder was specially designed and printed on a 3D printer. The micrometric screw and the load cell are mounted on movable holders that move vertically on to leading stainless steel rods. The load cell holder is equipped with linear bearings and moves freely, while the micrometric screw holder can be tightly fixed to leading stainless steel rods using screw clamps. Due to that, the device can be used for both 0.5-L and 0.33-L cans. The load cell can measure forces up to 100 N and is equipped with a hemispherical punch with a diameter of 15.5 mm. The diameter of the punch is large enough to prevent the wall of the can from being destroyed when the force on the can increases. The hemispherical punch is removable.

During the measurement the micrometric screw holder is tightly fixed to the rods. The head of micrometric screw presses the freely movable load cell holder in a defined manner and the hemispherical punch pushes the can. The punch deflects the can in half of its length perpendicular to the axis and to the surface of the can. The load cell is equipped with DMS meander-formed wires that change its resistance with the load. There are four resistors in the cell arranged in Wheatstone bridge. The bridge is interfaced using a precision 24-bit ADC for

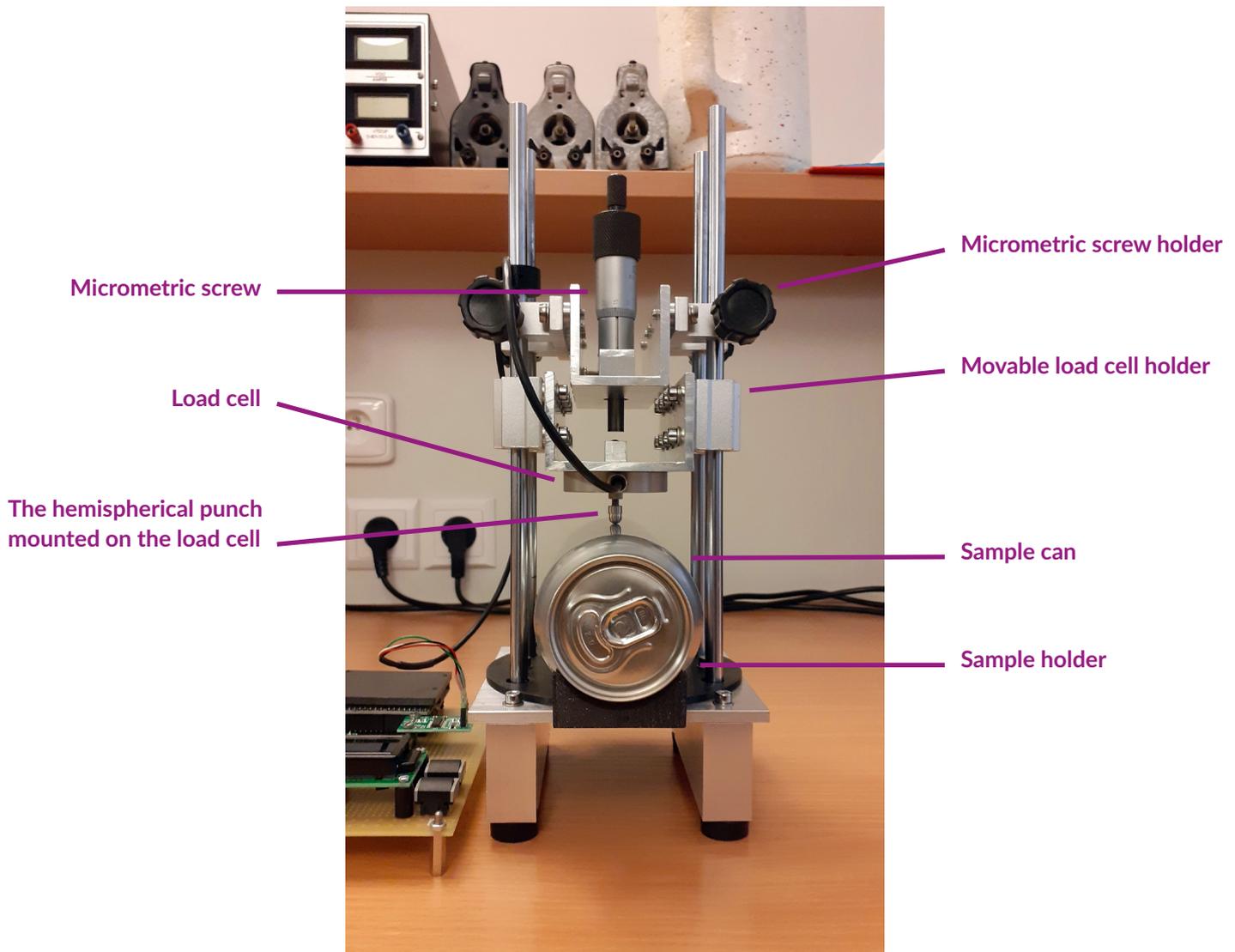


Figure 1 Device for the measurement of force –displacement curves with sample can

scales H711 (Avia Semiconductors) what enables force measurement with the resolution better than 0.001 N.

The force from the load sensor is logged by an 8-bit microcontroller Arduino Uno Rev3. The microcontroller simultaneously measures ambient temperature, calculates the slope of the force-displacement curve and provide a simple user interface using a display and 2 keys. Additionally, the data can be sent to a PC and the controller enables a calibration of the load cell using a weight with known mass.

2.7 Measurement of force-displacement curves

Before the measurement, sample cans were left to equilibrate for 24 hours at room temperature. The can was placed in the holder. The movable load cell holder was placed freely on top of the can, the micrometric screw holder was fixed at the top of the load cell holder. The micrometric screw was slowly moved against the load cell holder until the force applied to the can reached 5 N. The position of the micro-

meter screw was set as 0 mm displacement value. Then the position of the micrometric screw was gradually moved manually with the step of 0.05 mm and the force values were recorded. Maximal displacement was 0.2 mm, hence, each measurement consists of 5 points. The slope of the dependence of force on displacement $dForce/dDisplacement$ (N/mm) was calculated by the method of least squares.

3 Results and discussion

According to the instructions in the methods, samples with CO_2 concentration from 0 to 7 g/L were prepared. Used aluminium cans have a declared maximum safe pressure of 690 kPa (6.9 atm) (Kegland, 2023). Cans pressurized with dissolved CO_2 were used for the measurements, as this was the simplest way to create samples with a defined internal pressure.

At a room temperature of around 25 °C, with a CO₂ content of 7 g/L, the pressure in the can reaches 450 kPa (4.5 Bar). With increasing temperature, the pressure in the closed can increases rapidly and the maximum safe pressure of 690 kPa is reached already at 39 °C. The standard CO₂ concentration in beer is in the range of 4.5 g/L to 5.5 g/L. Therefore, from the point of view of safety, both during preparation, measurement and storage of the sample, the maximum CO₂ concentration of 7 g/L was chosen, which is higher than the maximum concentration used in the carbonation of beers.

Figure 2 shows the typical force-displacement curve measured on the aluminium can with concentration of 4 g/L CO₂. The pressure increases with the displacement of the can wall, the curve is slightly convex and can be fitted with a second-order polynomial curve with a great accuracy.

The non-linearity of the measured curve is small so the measured points in the displacement range of 0.0–0.2 mm are linearly correlated with the squared correlation factor value of 0.9997. All following measurements were made in a micrometer displacement range of 0.0–0.2 mm.

To verify the reproducibility of force-displacement measurements the can with a concentration of CO₂ 4 g/L was measured twelve times at the same position of the can. Figure 3 shows the first three measured datasets. The force-displacement curves were fitted with a linear curve and slopes dForce/dDisplacement (N/mm) were calculated. The linear dependence fits the curves with great accuracy, squared correlation factor of all calculated fits is better than 0.9998. The temperature of the sample changed by less than 0.2 °C during the measurement.

Figure 4 shows the calculated slopes of force-displacement curves measured at the same position of the can. Interestingly, the very first measurement result differs from all other subsequent measurements. The force displacement slope is lower in about 3.5%. There was no

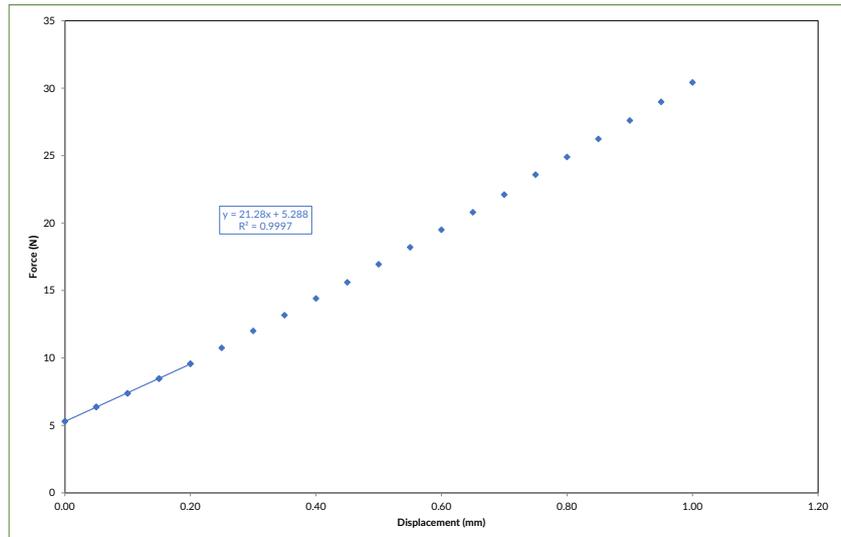


Figure 2 Force-displacement curve measured on the aluminium can with concentration 4 g/L CO₂

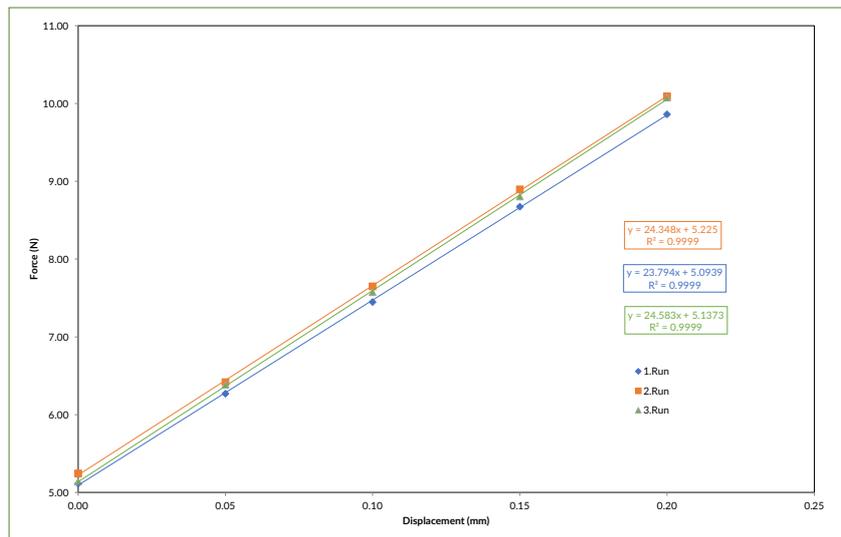


Figure 3 Force-displacement curves measured on the aluminium can with the concentration 4 g/L CO₂ in the range of displacement 0.0–0.2 mm

load on the can before the first measurement for several hours. The time difference between all other measurements was from 1 to 5 min. We received the same result when we waited two hours and repeated the same set of measurements on a different spot on the can. A possible explanation is that there is a process in the can with long relaxation. As a result, the following measurements were carried out at least twice and the first one was discarded.

The relative standard deviation of the force-displacement slope is from 0.6 to 1.0% for all measurements. We calculated the average value of dForce/dDisplacement slope from the second to the twelfth measurement. The average is 24.65±0.18 N/mm. The measurement of the force-displacement curve in one spot of the can is repeatable and reproducible with a relative standard deviation of 0.8%.

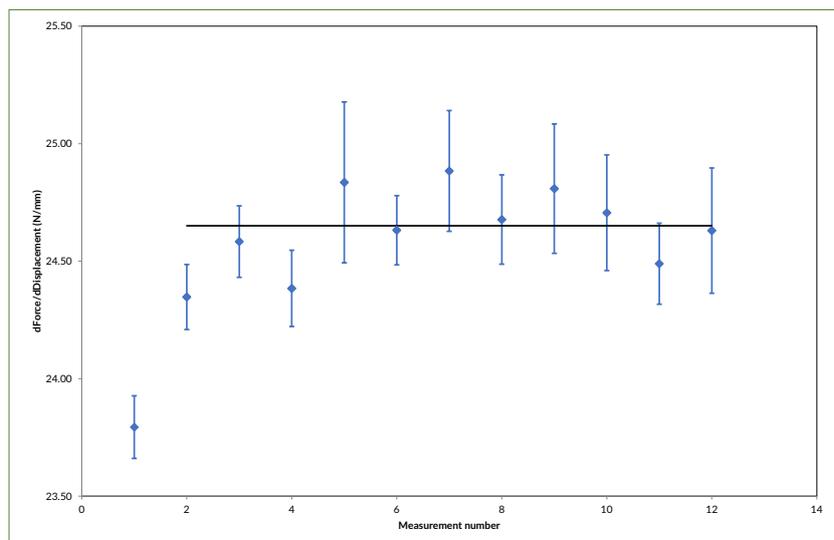


Figure 4 Slopes of force-displacement measurements measured at the same position of the aluminium can with the concentration 4 g/L CO₂ in the range of displacement 2 mm

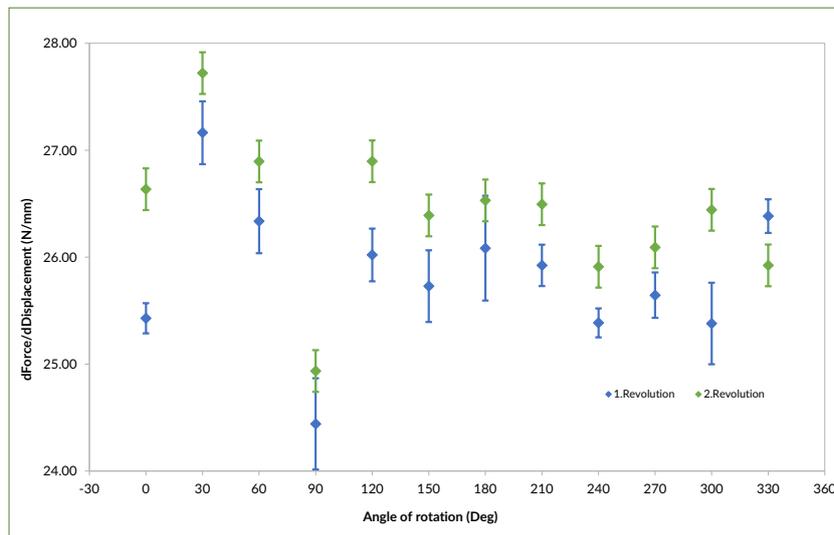


Figure 5 Dependence of the slope of force-displacement curve on the can position, aluminium can with concentration 5 g/L CO₂

We tested how the position of the can will affect the force-displacement measurement. The can with a concentration of CO₂ 5 g/L was rotated in the device with a step of 30°. In this manner, 12 measurements on the revolution were done. The measurement along the whole revolution was repeated two times. The resulted dForce/dDisplacement (N/mm) slopes can be seen in the Figure 5. The shape of the curve is identical for both turns. It became obvious, that there are local inhomogeneities of the can's tin causing variation in the results. The relative standard deviation of the dForce/dDisplacement (N/mm) slope in one revolution is 2.5% which is three times worse than the standard deviation of the measurement at the same place.

The average dForce/dDisplacement (N/mm) slope in the second revolution is higher by 0.6 N/mm when compared to the first one. This is caused by temperature

because each revolution was measured at slightly different temperature. The internal pressure in cans is dependent on the ambient temperature and increases with increasing temperature. During the measurement of the second revolution, the can temperature was higher by 0.6°C and higher dForce/dDisplacement slope values correspond to this fact.

Force-displacement curves were measured on prepared cans with CO₂ concentration from 0 to 7 g/L on the developed device. Before the measurement, the cans were left to equilibrate for 24 hours at a room temperature. Each can was measured 6 times, between two measurements the can was rotated with a step of 60°. The force-displacement curves were linear fitted and the slope dForce/dDisplacement was calculated. The average and standard deviation of resulting slopes of all 6 measurements was evaluated. The relative standard deviation of the calculated slopes was from 2% to 4% of the value.

The dependence between the slope dForce/dDisplacement and the concentration of CO₂ in cans is plotted on the Figure 6 separately for all three measured sets of samples. For better clarity of the figure, error bars are shown only for sample set 1. The dependence

is almost linear; however, the slope for the lowest concentration of 1 g/L CO₂ deviates substantially from the linearity. As shown on the Figure 6, the relationship between the dForce/dDisplacement slope and CO₂ concentration is linear starting from 2 g/L of CO₂ (with squared correlation factor higher than 0.991). The possible explanation is that the contribution of the can wall to the slope dForce/dDisplacement value is constant starting from 2 g/L CO₂.

The fitted lines are almost parallel for individual sample sets, but shifted with small offset. This might be caused by temperature dependence of internal pressure in the can. Each sample set was measured on a different day and the temperature in the laboratory varied slightly. The internal pressure in the can increases with increasing temperature. Therefore, the fitted line offset is higher for higher ambient temperature.

The force-displacement test responds to the internal pressure in the can. The pressure in the can depends on the concentration of dissolved CO₂ and increases significantly with rising temperature (Speers and MacIntosh, 2013). At the usual concentration of CO₂ in Pilsner-type beer of 5 g/L (Kosin et al., 2018), the pressure in the can increases from 56 kPa (0.56 atm) at 0 °C to 209 kPa (2.09 atm) at 20 °C, more than four times. It is therefore important to maintain a defined and constant temperature of the sample during the measurement. A change in temperature by 1 °C causes a change in the internal pressure in the can from 3 to 5%. dForce/dDisplacement (N/mm) results obtained from the force-displacement curves are more suitable to be displayed as a function of the internal pressure in the can rather than the CO₂ concentration.

The partial pressure of CO₂ was calculated from the CO₂ concentration and actual temperature according to Henry's law with the parameters determined by the National Institute for Standards and Technology of U.S. Department of Commerce (NIST) for aqueous solutions of CO₂ (NIST, 2023). The effect of acid was neglected. Figure 7 shows the dependence of the slope of force-displacement curve (dForce/dDisplacement (N/mm)) as a function of the internal pressure in the can for all three sets of cans. As expected, the points are more compact and following one line, however, there is still an effect of the ambient temperature. This might be an effect coming from the load cell and/or from the electronics. These effects have not been described yet.

Results obtained on prepared aluminium cans were compared to a sample of commercial aluminium can with lager beer Pilsner Urquell. Commercial beer can was measured ten times at the same position of the can. The relative standard deviation of the slope dForce/dDisplacement was 0.7%. This result is comparable to the measurement repeatability of the prepared aluminium cans. However, no difference between the first and subsequent measurements was observed. Like the prepared aluminium can, the commercial can with Pilsner Urquell was rotated in the device with a step of 30°. The measurement along the whole circumflex has been repeated two times. The result can be seen on the Figure 8. The shape of the curve is identical for both revolutions. The relative standard deviation of the slope in one revolution is 1.2% which is two times better

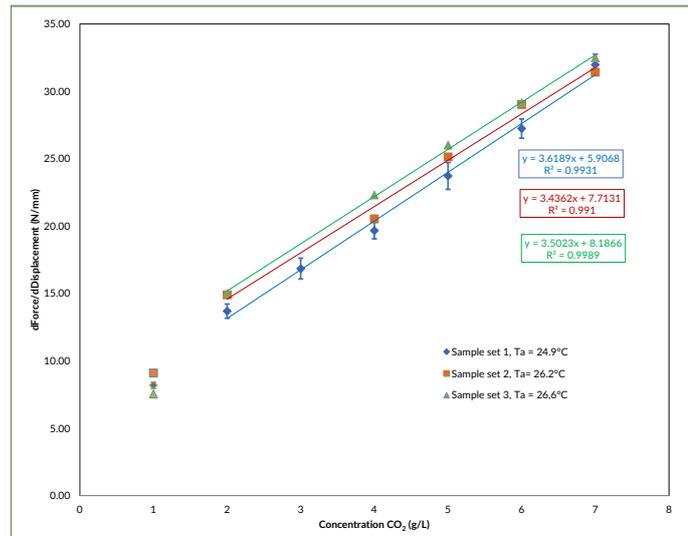


Figure 6 Dependence of the slope of force-displacement curve (dForce/dDisplacement (N/mm)) on the CO₂ concentration in the can

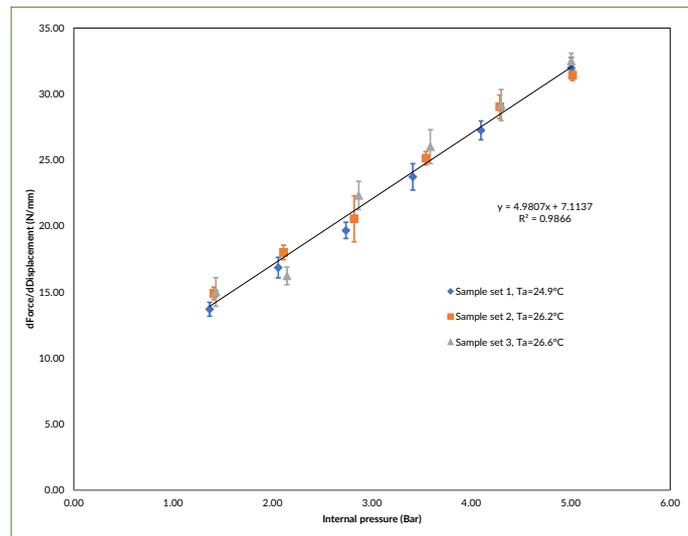


Figure 7 Dependence of the slope of force-displacement curve (dForce/dDisplacement (N/mm)) on the internal pressure in the can

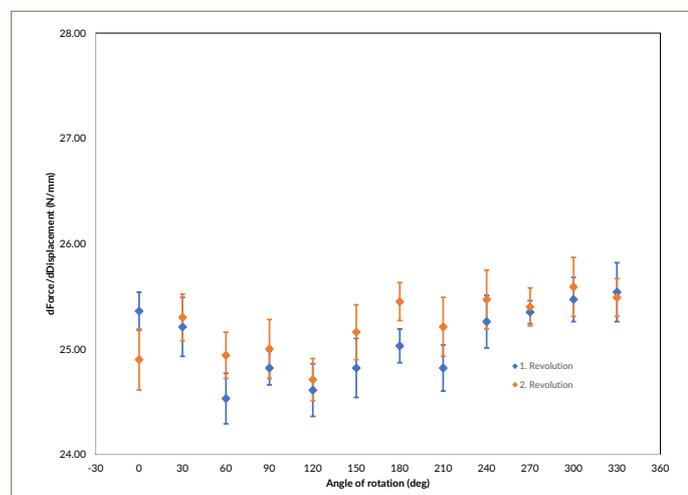


Figure 8 Dependence of the slope of force-displacement curve on the position of the steel can filled with beer Pilsner Urquell

when compared to prepared aluminium cans, but still worse than the standard deviation of the measurement at the same place. The measured commercial can seems to be more homogeneous compared to prepared aluminium cans.

All measurements with commercial can have been made at 26.5 °C. This is the same temperature at which the Set 3 of prepared aluminium cans was measured. Using the Set 3 for calibration, the concentration of the CO₂ in Pilsner Urquell can be determined. The mean slope for Pilsner Urquell was 25.20±0.27 N/mm, which results in 4.78±0.05 g/L CO₂ concentration. The CO₂ concentration in the can has been determined by Carbo QC from Anton-Paar, GmbH with the result 4.86±0.05 g/L CO₂. The result corresponds excellently with the result measured by the device.

The specifications of the developed device derived from the measured data are listed in Table 1.

Low repeatability of the developed device is not caused by the hardware or the detector, but is a consequence of the inhomogeneity of cans. There is quite high variability between materials used among various can suppliers, which influences wall thickness. Also embossing on can wall design, or different lacquer can influence the results of force-displacement measurement. To verify the influence of cans on the measurement results, it is necessary to carry out more tests on commercial samples. In order to be able to perform a larger number of measurements, it is planned to fully automate the device by installing an automatic motor-driven displacement of the micrometric screw and automatic turning of the can. The internal pressure in beer can and the result of force-displacement measurement is strongly dependent on temperature. To obtain the most accurate results it is necessary to store the samples at the exact temperature in a thermostated area.

4 Conclusion

We have developed and tested device for non-destructive monitoring of the internal pressure in a beer can based on the measurement of force-displacement curves. The measured force-displacement curves were linear and their slopes increase linearly with the internal pressure in the can. Low repeatability of the force-displacement measurement in cans does not allow its use for the exact determination of the absolute CO₂ content. The non-destructiveness of force-displacement measurement enables to use the device for monitoring the internal pressure changes in cans, for example in storage. In storage, the cans are deposited at a constant temperature and CO₂ in the liquid and headspace are in equilibrium. Fouling processes in the can are accompanied by pressure increase in time. Since the temperature in storage

Table 1 The specifications of the device for measuring force-displacement curves on beer cans

Parameter	Specification	Remark
Range	0.5–5 Bar (tested)	to 7 Bar (not tested)
Resolution	0.1 Bar	95% confidence
Repeatability	0.25 Bar	95% confidence

is constant, the temperature effects of the sample and the device can be neglected. If the device is used for time dependent analysis of the same can from the storage, accuracy can be increased by measuring on the same spot of the can.

5 Acknowledgement

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