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Ruggedized Color Measurement for Beer, Wort, and Malt

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ABSTRACT

The standard instrument for measuring malt, wort, and beer color is the spectrophotometer. Spectrophotometers are not rugged; they have critically aligned collimators, monochromators, lenses, slits, and mirrors that make them difficult to use and maintain in a production environment. Our research shows that wort and beer color can be measured accurately with rugged equipment based on light emitting diodes (LEDs) with results in agreement with those of a spectrophotometer. Two or more colored diodes were used. LED-based apparatus does not require critical alignment, is not highly sensitive to environmental conditions such as temperature, moisture, dust, and vibration, and can be rapid, inexpensive, compact, rugged, and easy to use. Statistical comparison in the range of 1–9 °SRM shows an insignificant bias for (SRM–LED) of −0.06 ± 0.4 °SRM. The intraclass correlation coefficient for the differences was 0.9952.

KEYWORDS

Bland–Altman; color; spectrophotometer; LED; SRM

Introduction

Beer and wort color are usually described by the absorbance at 430 nm. The color scale specified by the American Society of Brewing Chemists informally called the Standard Research Method (SRM), officially designated Beer-10, is defined by 10 times the absorbance at 430 nm of a clear (turbidity-free) sample of beer with a light path length of 0.5 inch or 12.7 times the absorbance in a sample with a path length of 10 mm.[1] The European Brewing Convention (EBC) specifies 25 times the absorbance at 430 nm through a 10 mm sample.[2] The SRM and EBC systems are used to describe the color of finished beer, of beer wort, and malt. Malt color refers to the color of wort prepared from a malt sample by a standard procedure.[3] The use of a measurement at a single wavelength has well-known limitations, but it serves well as a quality indicator for many purposes.[4] In a spectrophotometer, white light from a source (usually an incandescent light bulb) is collimated with lenses and mirrors, enters a monochromator through a slit, is dispersed on a diffraction grating, and the light of the selected wavelength exits through another slit. The width of the slits determines the band width of the spectrometer. For color measurement by ASBC Beer 10, a band width of 1 nm or less is specified. The monochromatic light passes through the sample and then to a detector. The angles of the incident and diffracted beams and the spacing of the grooves (rulings) on the grating determine the wavelength of the light that exits the monochromator. The wavelength is chosen by rotating the grating. The alignment of the parts of the monochromator, as well as the mirrors and lenses that direct the light from the source to the monochromator, from the monochromator to the sample, and then to the detector, are critical. Spectrophotometers are not well suited to environments with vibration, temperature changes, moisture, and dust; they are not appropriate technology for use on the production floor of a brewery or malt house.

A filter photometer is an alternative to a spectrophotometer. Light from the source passes through an optical filter that transmits at the desired wavelength. Filters usually have a pass band of around 50 nm. The filter has no moving parts and its alignment is not critical. Because the amount of light absorbed by a beer or wort sample is not a linear function of the wavelength, as shown in Figure 1, the range of wavelengths transmitted by the filter affects the result. This can introduce errors into the reading, so filter photometer readings for each brewery product must be compared with spectrophotometer readings to establish the relationship between the photometer output and the standard spectrophotometer result. This tedious and expensive process is described in the ASBC Methods of Analysis Beer 10B.[1] Little advantage is derived from this approach unless the company has multiple photometers and a central spectrophotometer. Facilities that produce many products, such as most craft breweries, would find photometer calibration procedures particularly disadvantageous.

Spectrophotometers and photometers have incandescent light sources that typically require 10–50 W or more of electrical power. A type AA battery can provide about 1000 mAhr = 9 kJ of electrical work. A 10 W source will exhaust the battery in 900 s (15 min). Incandescent sources generate heat that must be removed as they operate at a high temperature. Until the source reaches a steady temperature, the light output varies, so there is a significant warm-up time.

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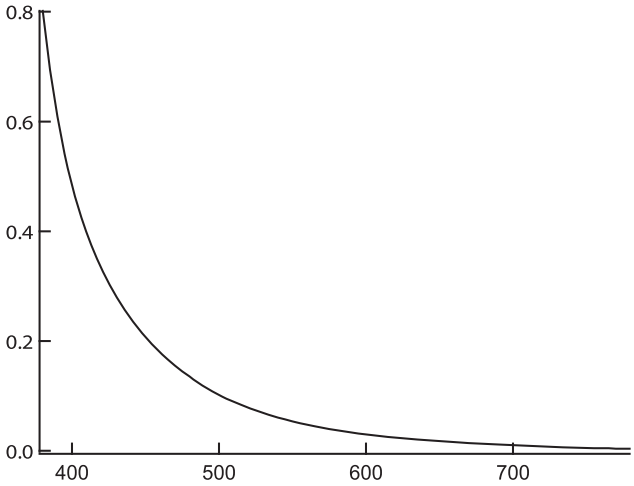

Figure 1. Spectrum of ale malt wort prepared in the laboratory.

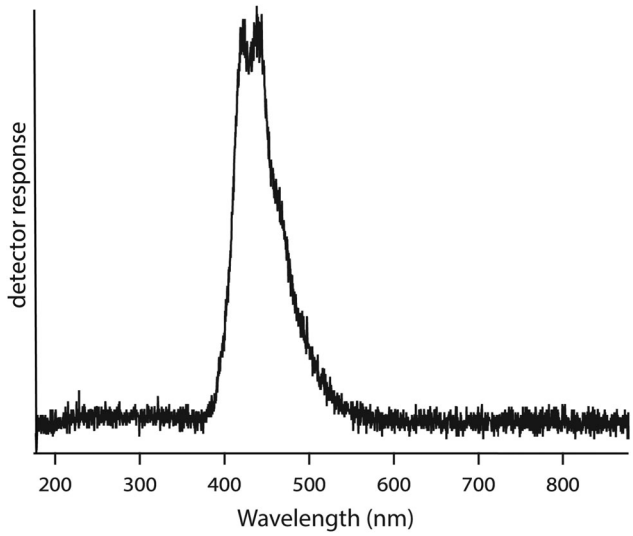

Figure 2. LED emission spectrum.

These issues limit the extent to which these devices can be made fully portable.

An alternative to using a white light source and a filter is to use a light-emitting diode (LED) as a source. Typical light-emitting diodes have an emission spectrum roughly complementary to the absorption spectrum of a filter. Figure 2 shows the emission spectrum of an LED whose nominal wavelength is 430 nm.

Although there is no approved beer color method involving an LED as a source, a device based on an LED would be expected to have performance similar to that of a white light source and a filter. No filter or monochromator would be needed. The power requirement for an LED is typically of the order of 0.1 W, which can be easily provided by conventional batteries. The LED coupled with a photodiode detector is rugged, stable, generates little heat, is inexpensive, and, lacking the bulky power supply and heat dissipation requirement of incandescent bulbs, can be made small and light. No warm-up time is required. These factors match the demands of a production facility. Because the LED emission band is much broader than the 1 nm specified in Beer 10,[1] the same time-consuming process of developing a specific calibration for each product as with a filter photometer would be required.

We have developed a technique of using two LED's with different central wavelengths to measure the sample absorbance. The absorbances at two wavelengths were fitted to the absorbance measured in a spectrophotometer with a 1 nm spectral bandwidth. A single fit gave satisfactory results for a variety of wort and beer samples. This method has all the advantages of an LED photometer, but without the disadvantage of tedious calibration for each brewery product. A calibration applicable to any sample could be performed at the factory, so the brewery or malt house could put the device directly to use. The LEDs consume little power, so they and their power supplies dissipate little heat. These factors allow the device to be rugged, light, compact, and portable.

Experimental

Wort

A variety of wort samples were prepared from various types and mixtures of commercial malt, including pilsner malt, ale malt, Vienna malt, Munich malt, and mixtures of ale or pilsner malt with chocolate malt and ale malt with caramel malt. Malt samples were a gift of the Briess Malt and Ingredients Company.

Worts were prepared by an accelerated microwave procedure.[5] After the malt was pulverized in a Procter-Silex coffee mill (blade mill), 25 g of grist (50 g in some cases) was added to a 500 mL culture flask (conical flask with screw-on lid); 400 mL of deionized, reverse-osmosis water was added, the flask was covered and shaken; the lid was removed and the flask was subjected to treatment in a microwave oven (Danby model DMW077BLSDD) whose nominal power rating was 1050 W. After treatment for 2 min at 90% power, the cover was replaced; the sample was shaken, the cover was removed, and the sample was subjected to an additional 1.5 min of microwave treatment at 90% power. The sample attained a temperature of 72 °C. All wort samples were filtered hot through Ahlstrom type 509 fluted 320 mm diameter filter paper via Mooney funnels, then driven through 0.45 μm pore size nylon membrane syringe filters (Pall). This method is used as a fast way to generate barley wort samples for validation of color measurements. It is not intended as a substitute for ASBC Method Malt 4.[3]

Beer

Eight commercial beer samples: Lagunitas Brown Shugga, Rolling Rock, Troegs Troegenator, Lagunitas A Little Sumpin, Heineken Light, Victory Prima Pils, Guinness Stout, Magic Hat #9, Guinness Extra Stout, and Victory Hop Devil were decarbonated on a magnetic stirrer (~15 min), diluted with deionized water if necessary, then tested for color.

Some wort and beer samples were diluted with deionized water to bring their absorbances into the measurement range and to generate additional samples.

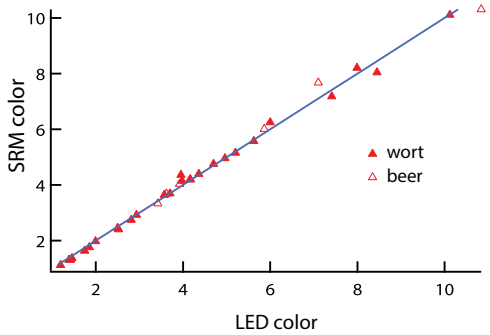

Figure 3. Comparison color by beer 10 (SRM color) with color by LEDs.

Absorbance measurements

Samples were measured in a MicroLAB® (Bozeman, MT) model 522 laboratory measurement system equipped with 16 colored LEDs and 8 photodiodes. The LEDs are mounted recessed into wells around the perimeter of the cylindrical sample compartment with photodiodes in wells on the opposite wall of the compartment. For Figure 2, the output of the 430 nm LED was directed via a fiber optic to a diode array spectrometer (Ocean Optic). The sample was held at the center of the compartment in a cylindrical glass vial whose internal diameter was 20.5 mm. The same samples were measured on the same days in 10 mm path length square glass cuvettes on a Cary model 300 double-beam grating spectrophotometer set for a band width of 1 nm. An identical cell with water was placed in the reference beam. A water sample in the same cell was run as a background measurement. These measurements were conducted on 36 samples of wort and beer. For each sample, the absorbance at 700 nm was used to determine if the sample was turbidity-free. Despite filtration through paper and 0.45 μm nylon filters, many of the wort samples and all the Guinness Stout samples did not qualify as "turbidity-free" according to Beer 10.[1] These samples were nonetheless included in the analysis.

Results

In these tests, a 400 nm and a 470 nm LED were used. The light intensity from each LED was measured with a photodiode first through a pure water sample, and then through the beer or wort sample and the absorbances were recorded. The same sample was also measured in the range of 715–400 nm on the spectrophotometer. The absorbances at 700 nm measured on the spectrophotometer were used to determine if the samples were free of significant turbidity. The absorbances measured at 430 nm on the spectrophotometer were used to calculate the color of each sample in °SRM as defined in ASBC Method Beer-10. These measurements served as the dependent variable. Ten samples randomly selected from those whose SRM values were 8 or less were used to generate the fitting parameters. The absorbances measured from the 400 and 470 nm LEDs served as independent variables. The relationship of the dependent variable to the independent variables was determined with a multilinear least-squares fit using the regression routine in Microsoft Excel®. The relationship took the form

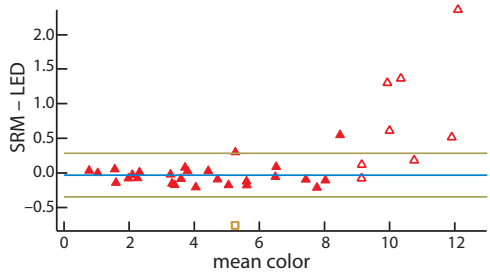

Figure 4. Bland–Altman plot SRM–LED. Empty triangular points have mean color greater than 9 °SRM and are excluded from the analysis. Square point is an outlier.

$$\mathrm{SRM} = a \times A_{400} + b \times A_{470} + c$$

where SRM is the color in °SRM, and A_{400} and A_{470} are the absorbances measured with the LEDs of the indicated nominal wavelengths. The constants a, b, and c are the parameters that were adjusted to minimize the sum of the squares of the deviations of the calculated values from the right-hand side of relationship with the measured values on the left.

Once these parameters are determined for a variety of beer and wort samples, the relationship can be used to determine the SRM color for any sample within the range of applicability of the relationship, that is, beer and wort samples of moderate color without unusual colored materials, such as fruit. Figure 3 summarizes the results, where SRM color is the color derived from ASBC method Beer 10 using the spectrophotometer, and LED color is the color calculated by inserting the absorbances measured from LED's into the above equation. The solid line shows the ideal fit if the LED color had been exactly equal to the SRM color ($y = x$ line).

Because a revised way to measure beer, wort, and malt color is being introduced, it is essential to evaluate the level of agreement between the spectrophotometer method (SRM) and the method we report here (LED). The accepted statistical procedure is to establish the range of agreement, which is the range of the method differences within which 95% of all measurement would be expected to fall,[6] and the intraclass correlation coefficient (ICC).[7] To use this information, one must consider what is acceptable agreement. In absolute terms, a color difference less than 0.3 °SRM is usually barely perceptible to the consumer, or in any event, difficult to measure. This may be too simple a criterion. Very pale beer could have a color as low as 2.5 °SRM. A deviation of as much as 1.0 °SRM would be manifestly out of specification. A deviation of 10% (0.25 °SRM = 0.02 absorbance units) would be marginally within the measurement capabilities of most instrumentation. By contrast, beer whose color is 40 °SRM or higher is perceived as black. The 10% difference between 36 and 40 °SRM is imperceptible in practice. It seems reasonable (or at least not ridiculous) to regard a range of agreement of ±10% as acceptable. We will evaluate agreement both in terms of absolute difference and relative difference. The relevant graphic representations are Bland–Altman plots. The x-axis in both cases is the mean color (SRM + LED)/2. Figure 4 is a Bland–Altman plot showing the absolute differences, SRM − LED, plotted against the mean color. The average difference, called the

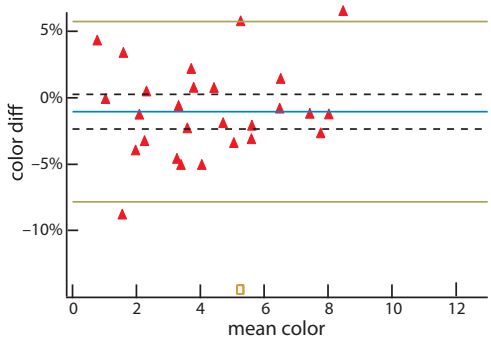

Figure 5. Bland–Altman plot (SRM–LED)/mean color. Out of range (> 9 °SRM) points omitted. Square point is an outlier.

Table 1. Agreement parameters.

	Lower limit of agreement	Upper limit of agreement	Bias
Absolute	−0.48 °SRM	+0.36 °SRM	−0.06 °SRM
Relative	−7.8%	+5.7%	−1.04%

bias, is marked with a blue line. The upper and lower limits of agreement are plotted as green lines. The 27 filled triangular points are those for which the mean color is 9 °SRM or less. The eight empty triangular points are those for which the mean color was greater than 9 °SRM. These points are regarded as out of the current range of the technique. They are excluded from the analysis. The square point was rejected because it is more than four standard deviations below the mean of the other in-range points. ICC is a commonly used statistic to measure agreement between bivariate measurements performed by separate devices or individuals. The ICC estimated using PROC MIXED in SAS version 9.4, for the data shown in Figure 4 was 0.9952 (95% confidence interval: 0.990–0.998). This is generally regarded as very strong agreement.[8]

Figure 5 presents the same data (out-of-range points excluded) but the differences are presented as relative to the mean color. Dashed lines show the confidence interval of the bias. Table 1 presents the agreement parameters. The Appendix is a summary of the measurements on wort and beer.

In this preliminary study it was shown that the limits of agreement of the accepted method for beer, wort, and malt color and a method based on LED measurements were within 0.5 °SRM or 8%, as summarized in Table 1. This level of agreement is satisfactory for most purposes. The major disadvantage is that the technique in its preliminary form is limited to samples whose color is no more than 9 °SRM. More highly colored samples can be diluted into this range. The factor that limits the range is the low transmittance of the samples at 400 nm. More work is needed to determine if the range can be extended and the limits of agreement contracted by varying the nominal wavelengths of the LEDs, especially the short wavelength LED, and by optimizing the light path length. It may even be possible to devise an apparatus with different path lengths for different LEDs. The apparatus on hand dictated the LED wavelengths and light path length in this preliminary work.

Conclusion

An LED-based device would be suitable for use under conditions in a production environment, such as a brewery or malt house, which can include humidity, vibration, and heat. An appropriate measuring apparatus should be sufficiently rugged to operate under these conditions without requiring frequent calibration, adjustment, or repair. Other desirable features include low demand for space and electrical power, portability, capability to be integrated into an in-line device, and digital connectivity. All of these features can be realized in an LED-based color-measuring device. The LEDs and photodiodes are typically around 6 mm in diameter and weigh less than 200 mg. The output from the photodiode can be amplified and digitized with readily available integrated circuits. A device could be made to fit in one's pocket and operate on batteries. Turbidity could be measured by including a photodiode offset from one of the LED's to measure scattered light. Another way to use an LED-based instrument would be to include it in the brewery plumbing to act as an in-line process sensor.

It seems likely that devices based on the same principles could be used for other common brewery measurements, such as beer bitterness (275 nm), diacetyl (530 nm), sulfur dioxide (530 nm), protein (250 and 275 nm), free amino nitrogen (570 nm), and many others. LEDs for wavelengths lower than about 350 nm are a great deal more expensive than those in the visible and infrared regions, but so too are the arc sources used in UV spectrophotometers. Such devices could be coupled to the effluent of a gas or liquid chromatograph and serve as selective detectors.

Disclosure statement

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Appendix: Summary of measurements on wort and beer

Sample	SRM	Absorbance 400 nm	Absorbance 470 nm	Turbidity-free
Yards IPA (beer) diluted to 10%	1.523142	0.429824	0.161878	
Heineken Light (beer)	3.240422	0.838584	0.338607	
Corona Familiar (beer)	3.55153	0.921113	0.35377	
Troegenator (beer) diluted to 5%	1.935088	0.488909	0.206424	
Rolling Rock (beer)	2.218164	0.584018	0.22403	
Victory Prima Pils (beer)	5.542323	1.498599	0.524421	
Pilsner	0.789125	0.208076	0.071998	no
Pale ale	1.58253	0.410689	0.143806	no
Vienna	1.034168	0.274547	0.100345	no
Munich	3.75702	0.943744	0.353534	
10% caramel 40° L in pale ale	3.257443	0.835214	0.318092	
10% chocolate in pilsner diluted to 20%	3.943088	0.892271	0.461545	no
10% amber	2.322267	0.594265	0.224	
10% caramel 80° L in pilsner	4.445987	1.100532	0.435175	
Pilsner 50 g	2.081185	0.556133	0.199493	
10% chocolate in pale ale diluted to 30%	5.527951	1.217811	0.635908	no
10% chocolate diluted to 40%	7.381314	1.566511	0.841945	no
10% chocolate diluted to 50%	9.204038	1.850835	1.041583	no
Lagunitas Brown Shugga (beer) diluted to 20%	4.667424	1.145631	0.483588	no
Lagunitas Brown Shugga (beer) diluted to 30%	6.56173	1.536673	0.663477	
Lagunitas Brown Shugga (beer) diluted to 40%	8.747414	1.845672	0.876381	
Lagunitas Brown Shugga (beer) diluted to 50%	11.0228	2.021763	1.08763	
Lagunitas Brown Shugga (beer) diluted to 60%	13.27359	2.110704	1.292967	
Pale ale	3.808456	0.940056	0.374491	no
10% chocolate in pilsner diluted to 20%	7.970515	1.658957	0.921612	no
10% chocolate in pilsner diluted to 12.5%	4.962066	1.086923	0.576492	no
10% chocolate in pilsner diluted to 8.3%	3.310124	0.738631	0.39219	no
Guinness Extra Stout (beer) diluted to 16.7%	12.16339	2.165279	1.410383	no
Guinness Extra Stout (beer) diluted to 12.5%	9.098175	1.836926	1.063843	no
Magic Hat no 9 (beer)	10.30029	1.972588	1.111336	no
Lagunitas Little Sumpn (beer)	6.459131	1.617201	0.641902	
Victory Hop Devil (beer) diluted to 50%	10.5798	1.979888	1.030832	
Guinness Draught Stout (beer) diluted to 10% filtered	5.41288	1.075188	0.576543	no
Guinness Draught Stout (beer) diluted to 10%	4.874345	1.175977	0.63686	no
Guinness Draught Stout (beer) diluted to 14.3%	7.662606	1.605397	0.903166	no
Guinness Draught Stout (beer) diluted to 20%	10.84395	2.047046	1.2674	no

Dark malts were mixed with paler malts. Example "10% chocolate in pilsner diluted to 10%" means that a sample of 2.5 g chocolate malt and 22.5 g pilsner malt was mashed and filtered. The filtrate was diluted to give 10% by weight of the original wort.