

THE DEVELOPMENT OF AN INSTRUMENT TO MEASURE COLOUR AND TURBIDITY ON-LINE IN REFINERY STREAMS

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Abstract

An instrument has been designed to measure colour and turbidity using solid state light sources and sensors operating at 470 nm (blue) and 880 nm (infrared) respectively. It is intended to be a factory instrument suitable for detecting break through from filters and colour gain or loss over sections of plant in a refinery. The measurements are expressed in absorbance units referred to a path length of 10 millimeters. A self-calibration feature is provided to zero the instrument on water. The optical cell, light sources and sensors are described. Results obtained by two instruments installed at Hulett's refinery are presented.

Introduction

There has been very little published on measurement of colour in refinery streams. The only reference is the work done by Plews *et al.* (1990) at Thames refinery where a photometer was modified to give a turbidity reading as well as colour. There are numerous methods to extract colour information from measurements made at 420 and 720 nm. Most of these involve subtracting the value of absorbance measured at 720 nm, multiplied by a factor, from the reading at 420 nm. The results varied considerably. A good summary is given by Carpenter (1983), in which it is stated that a basic requirement for the measurement of colour and turbidity is to be able to separate the two.

An instrument to measure colour as well as turbidity has been requested by the industry ever since the SMRI juice clarity meter (Stone, 1994) became available. Development in the field of colour measurement was dependent on the availability of a solid state light source with a wavelength near 420 nm (ICUMSA standard). A blue LED of 470 nm became available early in 1998. Although this is too far from the 420 nm standard for laboratory work, it was felt that it was close enough to enable a working instrument to be built that would give a relative indication of colour increase or decrease. No sample preparation is possible with an on-line instrument so it cannot produce results as accurate as the laboratory method for colour. The aim was to build a rugged instrument requiring minimum maintenance and suitable for detecting break through from filters and colour gain or loss over sections of plant in a refinery. The instrument also should require no attention other than routine cleaning about once a week.

Colour and turbidity measurement.

Colour measurements of sugar solutions are normally made by measuring the absorption of light at a wavelength of 420 nm after pH correction and filtration to remove all turbidity. Turbidity may be measured in a number of ways, e.g. light absorption, side scatter or forward scatter. The latter two methods are known as nephelometry. The most common method currently used is light absorption, although the use of nephelometry is being considered for low turbidity values of refined sugar solutions. Generally, light absorption is used for turbid solutions such as clear juice, whereas nephelometry is used for clear liquids such as fine liquor or drinking water. Forward scatter measurement has the advantage of being able to be implemented in such a way that the fouling of the cell window as well as colour is canceled out. However, since this instrument incorporates light absorbance measurement for colour for which no ratio method is available, it remains necessary to provide a water reference for calibration. Since this is necessary for the colour channel, the same reference can be used to zero the turbidity channel. Automatic calibration of an on-line nephelometer is very difficult because it requires the measurement of freshly prepared standard solutions or solid turbidity references. This greatly complicates the design if the aim of an unattended instrument is to be achieved. Also, although the refinery streams are generally fairly clear, it is not yet certain on which products this instrument would be used. Raw sugar melt, for example, is so highly turbid that it would be almost impossible to read using the same cell as fine liquor. These factors led to the decision that the best method for turbidity measurement is light absorption. It will be possible to modify the instrument later to use the nephelometer principle if this proves to be necessary.

Instrument design

Optical cell

The optical cell used in the juice clarity meter, from which this design evolved, was tubular. At the low brix levels of clear juice the defocussing effect of the refractive index of the clear juice was negligible. At the high brix levels of syrups the refractive index is so high that it is impossible to calibrate the cell on water. To avoid errors due to brix (refractive index) it is necessary for the light to pass through the cell windows at right angles. This meant that the cell had to be constructed with plane windows and the divergent light beam had to be collimated before entering the cell.

Light from the light emitting diode (LED) source diverges at an angle of 15 degrees. The spot on the LED from which the light emerges is small enough to be considered a point source, so if this is placed at the focus of a convex lens a reasonably parallel beam of light is produced. Similarly on the detector side of the cell, where the light emerges still as a collimated beam, a convex lens is used to focus the beam on the sensitive area of the detector. Using this optical system it is possible to move the LEDs and sensors away from the hot liquid so that no forced air cooling is required. It is also possible to seal the sensors completely thus making them insensitive to ambient light.

The flow-through optical cell is machined out of a single block of Ertalyte, a plastic material that can withstand temperatures of 120EC. The liquid flows upwards through a 24 mm threaded hole connected to the piping. Windows are let into the sides to accommodate the light sources and sensors. The distance between the windows (sample path length) is 28 mm. They are glued in place and then further clamped in place by the lens holder assemblies. It is possible to make a shorter path length version with windows 12 mm apart for darker solutions.

Light source

The monochromatic light required for the colour and turbidity measurements could be obtained in a number of ways. The conventional method used in a spectrophotometer uses a filament lamp and monochromator together with a fairly complicated optical system. Other fixed wavelength photometers use a filament lamp with filters of the required wavelength. These were all considered too delicate or required too much maintenance for use in a factory environment. The ideal light source is a light emitting diode of the correct wavelength and of sufficient power. Red LEDs at 640 nm have been available for some time and were used in the juice clarity meter. Blue LEDs of sufficient power only became available early in 1998 at which time the design of this instrument became possible. The wavelength of the light is a characteristic of the material from which the device is made. In this case the closest approximation to the 420 nm wavelength used for the laboratory determination of colour was 470 nm. The spectral bandwidth of these diodes is 35

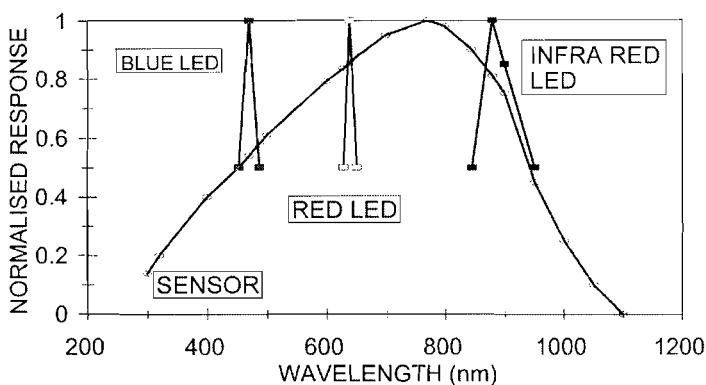


Figure 1. Spectral response of LEDs and sensor.

nm. Further development on these devices has produced one of 430 nm. Figure 1 shows the spectral response of the sensor relative to the wavelengths of the LEDs and their bandwidths.

Initially it was planned to produce results referred to the ICUMSA standard wavelengths but the nearest approximation to 720 nm, which was 640 nm, proved to be too close to the blue end of the spectrum and resulted in a turbidity signal with a fairly large colour component (see Figure 4). Since there are no LEDs available near 720 nm, it was decided to abandon this and choose a wavelength in the near-infrared. An LED of sufficient power was found at 880 nm. From the graphs in Figure 2, it can be seen that the absorption spectra of sugar solutions are fairly flat between 720 and 900 nm, so a light source at this wavelength would give very similar results to one at 720 nm but with the added advantage of being completely insensitive to colour.

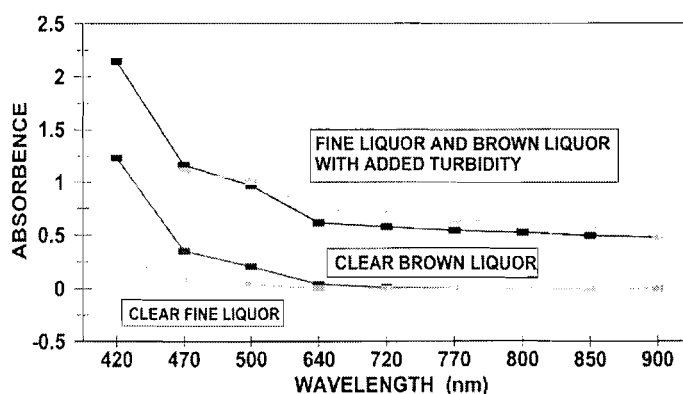


Figure 2. Absorption spectra of brown liquor and fine liquor.

Light sensor

Laboratory instruments normally use photomultipliers or photo diodes as light detectors. These give an analog output that has to be converted to digital form before being processed. The detector used in this instrument is a photodiode integrated with a current to frequency converter circuit on a single chip. The output is a five volt amplitude pulse train, the frequency of which is linearly proportional to light intensity. The dynamic range of the sensor covers six decades, which enables a very wide range instrument to be constructed which should be able to cover fine liquor as well as darker syrups. The pulse train, which is inherently a digital signal, can be counted very simply by a micro-processor to determine the frequency and hence the light intensity.

Mechanical

The mechanical layout of the instrument is shown in Figure 3. The prototype instrument has all the equipment mounted on a sheet metal baseplate and electrical connections are made via a cable and plug to a separate controller. Future models will have a smaller controller mounted on the same baseplate.

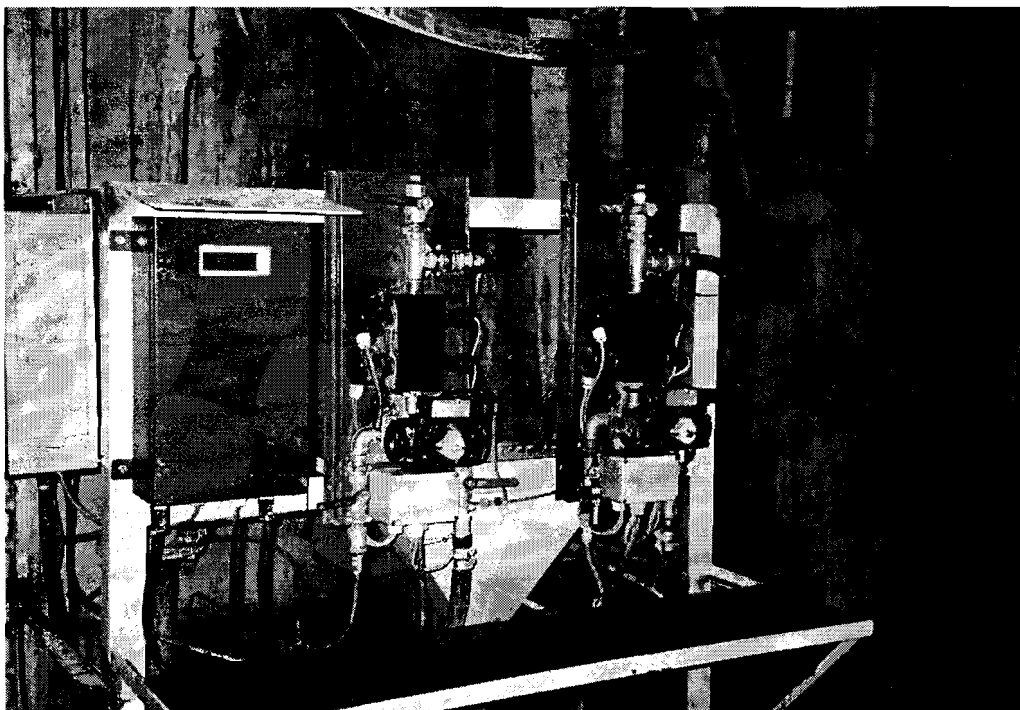


Figure 3. Two instruments and controller installed at HR.

The flow-through cell is mounted centrally and connected to a pneumatically operated change over valve. The valve selects the process liquid or calibrating water. It is operated once an hour by the controller to zero the instrument automatically. Manual valves are provided for safety to enable all incoming streams to be isolated. The output from the cell flows horizontally out of the discharge pipe. Vertically above the cell on the discharge pipe is a manual ball valve which enables the cell windows to be cleaned by inserting a bottle brush.

The photograph (Figure 3) shows two instruments and a controller installed at Hulett Refineries. The waste discharge can be seen directed into a tun dish. This discharge at atmospheric pressure is important so that no pressure is ever built up in the sample cell thus avoiding any possibility of damage or dangerous leaks of hot liquor. It also facilitates the collection of samples for checking calibration.

In a refinery the liquors are pumped between processes at fairly high pressures to accommodate the back pressure from filters and resin columns. Having this pressure available allows the instrument to be situated at any convenient place that will house it safely and provide a drain for the outflow to return to the process. In some cases the pressures are so high that the flow needs to be throttled. This is very difficult with manual valves because the setting of a partially closed valve changes with time. The connections to the factory piping were made with about two meters of 8 mm copper piping to slow down the flow through the instrument. This provided a secure connection while limiting the flow.

Controller

The functions of the controller are to:

- measure the sensor frequency and calculate the absorbance
- display the results locally and provide 4-20 mA outputs for remote indication
- carry out a self calibration at pre programmed intervals
- monitor the equipment for misoperation
- measure the degree of cell fouling
- check the absorbance levels against programmed set-points
- indicate alarms when abnormal conditions occur.

When the instrument is first switched on and it is known that the cell is clean, an initial calibration is carried out by a manual command entered on the keypad. The light intensity so measured is taken as the zero reference against which all further calibration readings are compared. At subsequent calibrations when the cell has become slightly fouled, the degree of fouling can be judged by comparison with the reference value. Fouling causing a light attenuation of up to 50% can easily be tolerated without degrading the instrument accuracy.

During normal operation the sensor frequencies are continually measured and the absorbance calculated as follows:

$$\text{Absorbance} = \text{Log}_{10} (\text{Reference frequency}/\text{Measured frequency})$$

where

Reference frequency = frequency obtained at the last calibration

Measured frequency = frequency obtained with process liquid in cell.

The results of the calculations for blue and red channels are displayed on the LCD screen as absorbance readings and transmitted as 4-20 mA signals.

Results

Two instruments were installed at the Hulett Refineries, one on fine liquor and the other on brown liquor. Initially the wavelength for turbidity measurement was set at 640 nm. The 4-20 mA outputs were recorded on a PC data logger. Figures 4 and 5 show the results obtained over two different 24 hour periods.

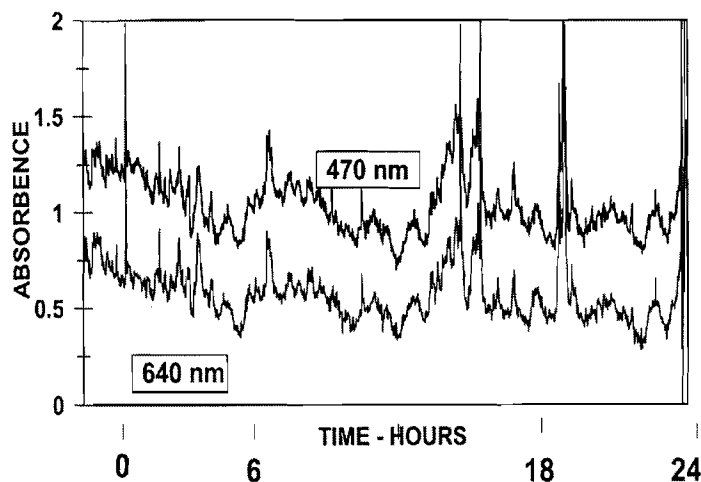


Figure 4. Results on brown liquor with 640 nm LED.

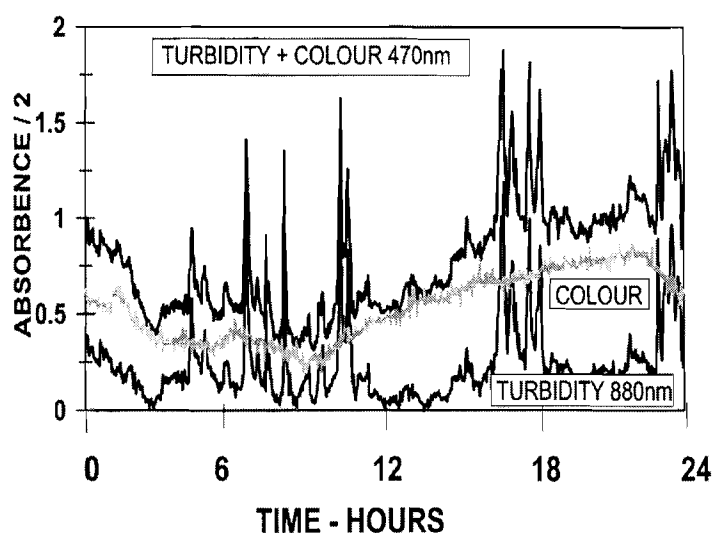


Figure 5. Results on brown liquor with 880 nm LED.

Results obtained on the brown liquor show that when measuring turbidity with a 640 nm light source (Figure 4), the colour has a considerable effect on the turbidity reading. The brown liquor appeared quite clear with no visible suspended matter, yet gave an absorbance reading of about 0,5 Abs. while the colour was about 1,6 Abs.

The 640 nm LED on the brown liquor channel was then changed to 880 nm. The results obtained are shown in Figure 5. It is now known from the spectra in Figure 2 that the tur-

bidity signal is independent of colour. The colour signal, however, still has extra attenuation due to turbidity added to it. If the turbidity signal multiplied by 1,1 is subtracted from the colour + turbidity signal the true colour trace is produced. From the graph it can be seen that the turbidity interference of the colour signal has been reduced to less than the natural scatter of the colour reading and is quite undetectable.

After a month's use at the refinery it was obvious that the compromise cell length of 28 mm could not accommodate the range of readings of fine liquor as well as brown liquor. The fine liquor readings ranged about 0,05 - 0,5 absorbance units while the brown liquor varied from 0,05 to over 4. This is a very large range which produces a light intensity change of greater than 10 000:1. The cell lengths will have to be adjusted to suit the absorbance of the product being measured.

The cell windows remained remarkably clean during these tests. This is probably due to the high temperatures and high brix inhibiting the growth of organic films and also to the absence of any lime.

Discussion

A problem remains as to what units to use for the display of colour measurements made by this instrument. Ideally ICUMSA colour units should be used. However this requires that the brix of the liquor be known and its pH adjusted. The wavelength at which the measurement should be made is 420 nm while that used in the instrument is 470 nm. The absorbance at 420 nm is about 3,5 times that at 470 nm. This could be incorporated as a correction factor in the calculation, but insufficient data has been collected to be certain that this factor is constant for all products.

The instrument is basically a dual wavelength photometer so the output could be expressed as the attenuation index but this also requires the concentration to be known. It is probably best to express the reading as an attenuation in absorbance units (A_{470} and A_{880}) referred to a path length of 10 millimeters so that the results can be compared with a laboratory spectrophotometer.

The accuracy of the instrument depends on a good calibration therefore it is important that the calibrating water is clear. Fine liquor has practically the same absorbance as water at 880 nm. At times it can actually be clearer, as evidenced by the occasional very slight negative readings on the turbidity. If there is any doubt about the clarity of the water supply, it is better to use condensate. This should be cooled to room temperature because the temperature of the outflow during calibration is used as an indication that the calibration water is flowing.

The results obtained at Hulett Refineries show many peaks of turbidity and colour which do not show up on the daily composite samples. The peak values are sometimes many times the average values.

Conclusion

An instrument has been developed and deployed in a sugar refinery where it is successfully indicating colour and turbidity of fine liquor and brown liquor. Results have been recorded for a month with no problems being experienced with the instrument. Colour trends and discrete turbidity incidents have been indicated with no interaction between the colour and turbidity signals. The results have been made available as a trend graph on a PC screen in the control room.

Acknowledgment

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