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Portable Instrument for the determination of Manganese in Steel

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Abstract: An opto-electronic instrument has been developed for the determination of Manganese (Mn) in steel. Manganese is an important element, which is added to steel in different amounts to improve mechanical properties. Method for the determination of manganese in steel is based on the permanganate violet color formation due to oxidation of Mn (II) to Mn (VII) having absorbance maxima at 525nm. The instrument consists of high intensity green colored light emitting diode (LED) of wavelength 525nm as light source. Photodiode BPW 21 as detector with spectral sensitivity above 90 % in the range 500-600nm has been used. Lambert-Beer's law is obeyed in the concentration range 0.5-20 ppm with path length of 1cm. Manganese in steel upto 2 % can be determined with the developed instrument.

1. INTRODUCTION

Steel is an alloy of iron and carbon that contains manganese as an alloying element. The alloying of steel with several metallic elements plays an important role for improving the mechanical and chemical properties such as strength, toughness, heat resisting, and corrosion resistance. In steel-making processes, the content of alloying elements such as manganese, chromium, nickel, molybdenum, vanadium etc, is required to be strictly controlled because it fundamentally determines the performance of steel materials. Manganese is usually introduced into steels to increase the tensile strength. Commonly, the concentration lies between 0.1% and 3% manganese by mass [1].

In the metal determination, several techniques employed such as neutron activation analysis, molecular absorption Spectrophotometry (UV-VIS), Neuron activation analysis, flame atomic absorption spectrometry, Electro-thermal atomic absorption spectrometry (ETAAS), Atomic emission spectrometry with inductively-coupled plasma excitation, X-ray fluorescence and Voltametry [2,3]. Usually such techniques need enrichment steps. However, molecular absorption Spectrophotometry is a very attractive tool for the analysis of steel samples due to its operational simplicity, low cost, high selectivity and automation potential.

In an attempt to overcome these limitations by the ease and simplicity of a direct determination, a portable instrument has been developed for the rapid and precise determination of Mn in steel. The instrument is designed to be compact, lightweight and rugged for field work. The difficulty with producing an LED based instrument in the past has been the limited choice of LEDs. Recent advances in LED technology have made such a device possible [4, 7]. The instrument discussed uses commercially available LEDs and other off-the-shelve electronic components which results in a low cost durable device. An advantage of this LED instrument is that its sensitivity is equal to or better than many research-grade or commercially available spectrophotometers and also includes low cost, compact size and high stability. These devices rely on the fact that a given LED produces light of a monochromatic wavelength associated with small range wavelength of light having maximum intensity at peak wavelength. However this inflexibility in wavelength can be considered a limitation as a physical exchange of the LED is usually required for a change of wavelength [8-10].

2.1 INSTRUMENT DESIGN

The instrument mainly uses an LED of 525nm wavelength (colour green) as light source replacing all conventional optics like grating, lenses, filter, mirrors etc and microcontroller (MCS-51) as the heart of the instrument. It compares the color of the resulting solution with the colors of a series of metal standards. The principle depends upon the fact that the percentage transmittance of light at a given wave length for a permanganate solution is a function of the manganese concentration by use of which the intensity of the light transmitted by a colored solution is correlated with the concentration of the constituent responsible for the color. This manganese concentration can easily be related to weight percentage in the steel sample. The instrument is adjusted so that the blank solution gives a reading of zero absorbance (or 100% transmittance); the colored solution then shows some absorption of the light.

The instrument works on the principle of Beer-Lambert's law. The linear region of absorption measurements can be described by the Beer-Lambert (Beer's) Law:

$$A= \varepsilon cl. \qquad (11)$$

where ε is the molar absorptivity (L mol^{-1} cm^{-1}),
l is the cell path length (1.0 cm for this experiment), and c is the concentration (mol l^{-1}).

The system consists of 89C51RD2BN, keypad, power supply, light source (LED), photo detector (BPW 21), ADC0804 and 16 x 2 LCD display. In current to voltage converter, an operational amplifier (op-amp) was used with a feedback resistor. ADC0804 chip had 8-bit resolution, which works on a +5v supply. In ADC 0804, conversion time can be controlled by using two external components resister and capacitor. A 16 x 2 digit LCD module was incorporated to display measurements. Keypad consists of four keys; one for switching LED ON/OFF and other three are used for percent transmission (%T), absorbance (ABS) and concentration (conc.). Fig. 1 shows the block diagram of the complete system:

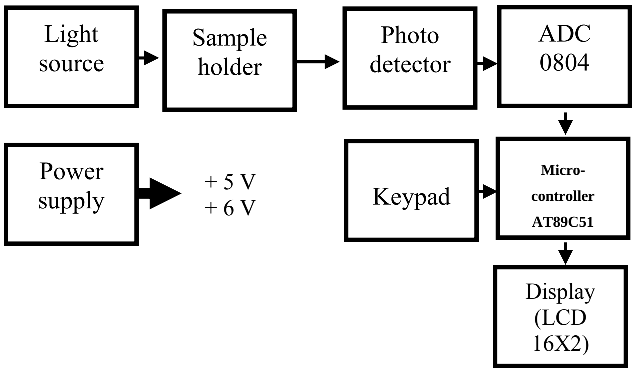

Fig. 1 Block diagram of the instrument

2.2 Power Supply

Single power supply of 6V & 4.5AH has been used for the source, detector, and current to voltage converter. The system has been built using rechargeable batteries to make ripple free supply and to maintain portability of the system.

2.3 Photo Detector (Bpw21)

A visible range photo detector is selected with operating voltage 6V and forward current 1.3mA (typ) and max 5mA, output current being 50mA. It is ideal for low light level applications where a very high signal to noise ratio is important. The relative spectral sensitivity of BPW21 varies between 0.9-1.0 at a wavelength from 500 - 600nm, which is very close to the human eye.

2.4 System Operation

A microcontroller-based system (Fig. 2) was fabricated to measure Mn concentration in Steel. The system is based on Lambert-Beer's law. Cuvette holder fabricated from Al alloy holds cuvette (path length, 1 cm), which holds LED at one end and detector system at the other. Test solution after color development is put in cuvette, which is placed inside cuvette holder. Absorption / transmittance, related to concentration, is detected by photodiode (current output in μA). A current to voltage converter converts microampere signal into millivolts / volts, which is changed to digital output by ADC0804 and is fed to microcontroller. Then software reads digital output. According to different solution concentrations, change in output voltage of I-V converter is noted and reference voltage of ADC is set. Microcontroller calculates ratio of two voltages (0% T, 100% T). Light intensity is varied to adjust zero setting. Software takes the ratio of sample with respect to blank solution (distilled water). Microcontroller takes input from ADC to calculate two ratios, which is then compared with look up table, which gives absorbance [Absorbance=2-log (%T)] and relates to concentration. This is displayed on a 16 x 2 LCD display. The programme flow chart is as shown in fig 3.

Fig.2 LED based developed Instrument

3. CHEMICAL ANALYSIS PROCEDURE

All reagents chemicals used are of AR/GR grade

Procedure

Weighed 0.1000 gm of steel sample into a 100 ml conical flask. Dissolved in 25 ml of 1:3 HNO_3 and boiled to expel nitrous fumes. Added 0.5-1.0 gm of ammonium persulphate and boiled for 10-15 minutes to oxidize any reducing agents present in the solution and to destroy excess of persulphate . If any permagnate colour develops or oxides of manganese separate, add a few drops of sulphurous acid or sodium sulphite solution to reduce the manganese and render the clear solution. Boil to expel excess of sulphur di oxide. Diluted the solution to about 50ml with distilled water. Added 5ml of syrupy phosphoric acid and about 0.5gm of potassium periodate. Boiled for 1 minute and allowed to cool to room temperature. Made the volume upto 100ml in a volumetric flask. Measured the absorbance value with the developed instrument having 1 cm path length cell.

$$\%\text{age of Mn} = \frac{0.0225 \times \text{Absorbance Value} \times \text{Volume of solution in ml} \times 100}{1000 \times \text{Weight of sample taken in gm}}$$

Analyzed standards and unknown steel samples with the developed instrument & spectrophotometer by adopting the above procedure and obtained the results as shown in Table 1. The weight of standard steel sample and unknown steel sample taken was 0.1000gm and final volume made after colour development was 100 ml.

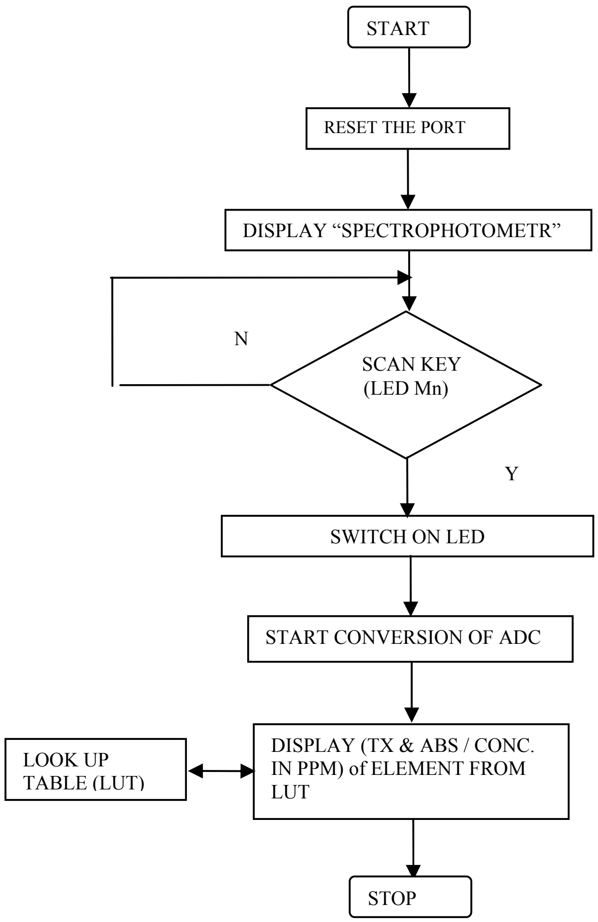

Fig. 3 Programme flow chart

4. RESULTS

The microcontroller reduces the circuitry and performs accurately. The results obtained with the ELICO SL-159 and the designed LED based Spectrophotometer is compared and is given in Table 1. The results obtained by the MCS-51 based optoelectronic system are accurate and as comparable to the available spectrophotometer (ELICO SL 159) as shown in Table 1.

Table 1 Comparison of results between ELICO SL 159 Spectrophotometer & Developed Instrument

Readings taken by ELICO SL-159 Spectrophotometer				**Readings taken by Developed Instrument**	
S. No.	Steel Sample / Standard	Absorba -nce Values	%age Mn	Absor-bance Values	% Mn
1.	BCS No. 407	0.058	0.13	0.061	0.14
2.	BCS No. 410	0.192	0.43	0.199	0.45
3.	Sample 1	0.308	0.69	0.306	0.68
4.	Sample 2	0.287	0.64	0.289	0.65
5.	Sample 3	0.143	0.32	0.147	0.33
6.	Sample 4	0.335	0.75	0.330	0.74
7.	Sample 5	0.518	1.16	0.522	1.17
8.	Sample 6	0.486	1.09	0.478	1.07
9.	Sample 7	0.412	0.93	0.410	0.92
10.	Sample 8	0.500	1.12	0.507	1.14

5. CONCLUSIONS

Optoelectronic components, such as light-emitting diodes (LEDs) and photodiodes (PDs), are robust, compact, low power consumption, long life and reliable compared to conventional incandescent or discharge lamps and photomultiplier tubes (PMTs). It is battery operated because these components feature low power consumption and high efficiency.

The development of portable system using LED as light source and microcontroller (MCS-51) has resulted in a more compact reliable and efficient measurement for this application. The use of LED reduces the use of filter / grating, lenses etc and the use of microcontroller eliminates the analog logarithmic stage. As the system is portable and battery operated, it is suitable for field applications.

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