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Microcontroller based portable instrument for determination of nickel in edible oils

Prasant K Mahapatra[1]*, Ashok Kumar[1], Mewa Singh[1], Narwant S Grewal[2], L Pandey[1] and M L Singla[1]
[1]Material Research Division, Central Scientific Instruments Organisation, Sector 30C, Chandigarh 160 030
[2]ECE Department, Guru Nanak Dev Engineering College, Ludhiana 141 006

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A microcontroller (μC) 89C51RD2BN based portable instrument has been developed for determination of nickel (Ni) concentration in edible oil using dimethylglyoxime method. An LED has been used as a light source with peak wavelength at 450 nm. Silicon photodiode is used as a detector having peak spectral response at 550 nm.

Keywords: Edible oil, LED, Microcontroller, Nickel

Introduction

Trace and transition metals should not be present in such quantities that endanger health or act as catalysts on oxidative evolution of oils and fats[1]. Quality of edible oils with regard to freshness, storability and toxicity can be evaluated by determination of metals. Trace metals (Cu, Mg, Ni, Fe) increase rate of oil oxidation while other elements (Cr, Cd, Pb) cause toxicity. Thus, development of rapid and accurate analytical instruments for trace elements determination in edible oil has been a challenge in quality control and food analysis[2]. Nickel affects respiratory tract (nasal cancer, asthma pulmonary fibrosis), skin, liver and heme metabolism. Among analytical methods[3,4] used for analysis of metal and non metal ions, spectrophotometers generally use a tungsten lamp as light source, an optical filter, detector, and display system etc[5].

This study presents design and development of a microcontroller-based portable instrument to determine nickel concentration in edible oil.

Materials and Methods

Instrumentation

System consists of 89C51RD2BN, keypad, power supply, light source (LED), photo detector (OSD-15E), ADC0804 and 16 x 2 LCD display (Fig. 1). A single power supply of +6V & 4.5AH for current to voltage converter and +5V from LM7805 regulator for the source, detector, microcontroller and ADC was used. Rechargeable battery was used to make ripple free supply and to maintain portability of the system. Light source was a super-bright blue LED[6] (wavelength = 450 nm, I_f =50 mA, V_f =4·8V, view angle = 16°). Photo Diode[7] (OSD -15E) was a visible range photo detector (operating voltage, 6V; wavelength range, 350 - 800 nm; active area, 15 mm^2; peak responsively, 550nm). 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[8]. A 16 x 2 digit LCD module was incorporated to display measurements. All active devices were contained on single CMOS integrated circuits (IC) including 7-segment decoder, display drivers, reference and clock. Seven segment drivers output were fed to 16 x 2 digits LCD for display[6]. 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.).

System Operation

A microcontroller-based system (Fig. 2) was fabricated to measure Ni concentration in edible oil. The system is

*Author for correspondence
E-mail: prasant22@yahoo.com

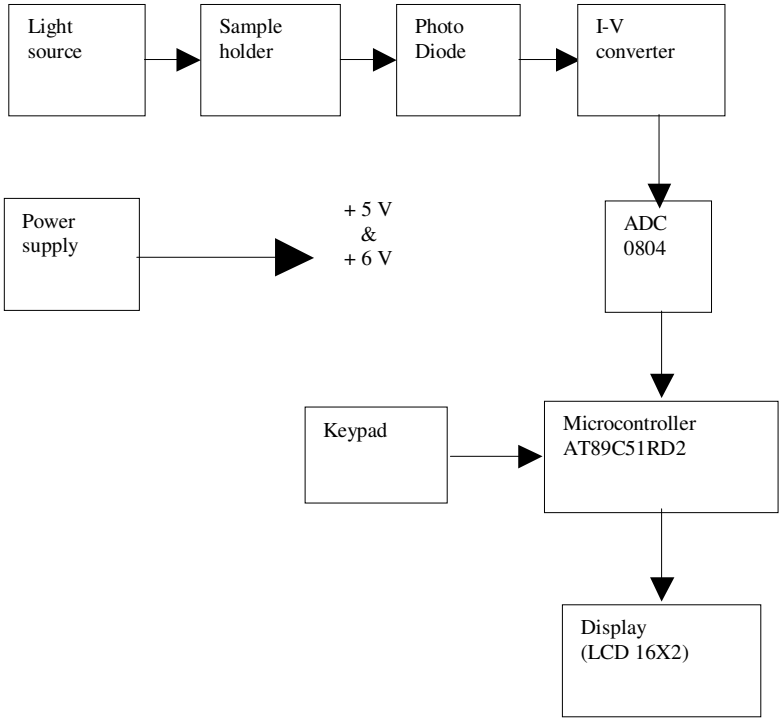

Fig 1—Block diagram of instrument

Fig. 2—Developed instrument

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 added 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 (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.

Analysis for Nickel in Edible Oils

Samples (1, 10 or 50 g) weighed into dish depending upon contents (for < 2 ppm, 50 g; for 2-10 ppm, 10 g; and for 10-100 ppm, 1 g). Samples heated gently until fat can be ignited by application of a small flame to the surface, followed by applying enough heat to keep the sample burning until residue becomes charred mass and then heated in a muffle furnace (550°C) for 1 h. Added 1-2 ml conc. HCl to dissolve residue. Further, heated gently to dissolve contents and then almost to dryness. Residue dissolved in distilled water and washed into 50 ml glass stoppered volumetric flask. Ni estimated as per test procedures.

Preparation of Standards and Reagents for Nickel

All chemicals used of AR/GR Grade. Dimethyl glyoxime (DMG) reagent (0.1%) was prepared by dissolving DMG (0.1 g) in ethyl alcohol (100 ml). For bromine water solution, distilled water (200 ml), added liquid bromine (2-3 ml) slowly until some liquid bromine remains undissolved at bottom. For standard Ni solution, nickel sulphate ($NiSO_4$ $7H_2O$, 0.4782 g) was dissolved in volumetric flask (100 ml) and diluted to get 2 ppm or desired Ni concentration.

To test solution (5 ml), bromine water was added drop wise until yellow colour persists, waited for 2 min, then added dropwise liquid ammonia until yellow colour disappears and added 1 ml in excess. Mixed and added 1 ml of DMG (0.1%) and made the volume upto 10 ml. Noted reading against reagent blank, prepared in similar way without Ni solution.

Table 1—Comparison of results between ELICO SL-159 UV-VIS spectrophotometer and microcontroller based portable instrument

S No.	Standard solution Ni conc. ppm	ELICO SL-159 spectrophotometer ppm	Developed instrument ppm
1	0.25	0.26	0.27
2	0.50	0.56	0.53
3	0.75	0.78	0.74
4	1.00	1.05	0.97
5	1.50	1.48	1.45
6	2.00	1.97	1.95

Table 2—Study of interference due to Fe & Cu

S No.	Ni conc. ppm	Fe added ppm	Cu added ppm	Developed instrument ppm
1	0.25	5	5	0.26
2	0.50	5	5	0.57
3	0.75	5	5	0.72
4	1.00	5	5	0.95
5	1.50	5	5	1.47
6	2.00	5	5	1.92

Results and Discussion

LED (450 nm) is specifically used to determine concentration level of Ni in edible oil. Microcontroller reduces circuitry and performs accurately. Designed instrument showed accurate results that are comparable to the available spectrophotometer (Table 1). Fe and Cu did not interfere as concentration values of Ni did not change in the presence of 5 ppm level of these metal ion impurities (Table 2). In case of Cr, Ni values decreased, indicating interference in this method. This was removed by adding 5% citric acid (1 ml) solution after adding Cr. This instrument can also analyze other metallic impurities just by changing desired wavelength LED.

Conclusions

Development of portable system using LED as light source and microcontroller has resulted in a more compact, reliable and efficient measurement system. Use of LED reduces use of various filters for different

elements, and use of microcontroller eliminates analog logarithmic stage. As the system is portable, it is suitable for field applications.

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