Experimental Investigations of On-line Fiber Optic H₂S Gas Sensor for Industrial Applications

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Fiber optic sensors to-date is a rapidly growing field with several potential benefits over the conventional electronic and electrical sensors. Intensity based fiber optic sensors are conceptually simple, ruggedised and cost-effective as they can be implemented using less expensive multimode fibers and their assemblies and other components. The various aspects of design, development and experimental investigations of an on-line fiber optic H₂S gas sensor based on interference free lead acetate detection technology for industrial flow lines are discussed.

Introduction

In several industrial and other applications such as for environmental control, catalyst protection, beverages and pharmaceutical manufacturing, it is essential to know the quantities of H₂S gas produced and total sulphur contents present in gas or liquid phases. Several electrical, chemical and electrochemical methods have been in use for determination of H₂S concentration 1-3. Optical sensing techniques in general are well-known for precise and non-intrusive measurements and in the recent years, there has been growing interest for the development of optical sensors for detection of toxic gases in environmental and industrial situations. In particular, fiber optic sensors offer the added advantages of ease and on-line/in-situ measurement, flexibility, geometric versatility, ruggedness, portability, immunity to electrical and electromagnetic interferences and resistance to corrosive and hostile environments 3-5. The technique being presented here involves the measurement of color change of lead acetate paper/detector (through reflectance of light) where a chemical reaction occurs between the lead acetate and H₂S gas sample flowing continuously to the detector. The chemical reaction results in the formation of lead sulphide which is measured as a brown stain on the detecting paper. This lead acetate technique is interference free and enables specific detection of H₂S both in gas and liquid phases.

Principle

This sensor operates on the principle of light being reflected off a lead acetate paper/detector and then received back through a receive branch of a bifurcated fiber optic bundle (Y-guide) on to a photodiode. Keeping the separation between the common end of the bundle and the detector surface as fixed, the quantity of light reflected back is directly governed by the concentration of H₂S which produces brown stains of lead sulphide after a chemical reaction with the lead acetate. The electrical signal displayed indicates the concentration of H₂S gas present from which the total sulphur contents can also be determined.

In the bifurcated fiber bundle developed and employed, one group of fibers transmits light from a stable source to a target reflector surface. The other group receives the light reflected from the target and transports it back to a photo-sensitive detector which converts it to

![Figure 1(a)-Arrangement of an optical fiber Y-guide (bifurcated bundle) used for reflection modulated fiber optic sensor.](image-url)
an electrical signal proportional to the intensity of the reflected light as depicted in Figure 1(a) while Figure 1(b) depicts the action of an adjacent pair of fiber optic light transmitters and receivers as used to detect the position and or quality/condition of a reflective surface relative to the ends of the optical fibers.

Evidently, as the reflecting surface moves away from the set of optical fibers, the area, A illuminated by the transmitting fiber optic element becomes larger and larger. The illuminated area of interface, B1, which is providing light to the surface of the receiving element as B2 also grows increasingly larger. There is a rapid and linear growth in the signal output as more of surface, C, is illuminated. This portion of the response curve is referred to as the "front slope region." The point at which the entire surface, C, becomes covered with light is referred to as the "optical peak" point of the response curve as shown in Figure 1(c). As the surface moves further away, the size of area B2 becomes larger than area C, reducing the detected light intensity. The light intensity-monitoring photosensitive detector output then begins to decrease, resulting in the back slope characteristic where the signal decreases proportional to the square of the distance between the probe and the surface.

The extremely rapid signal rise in the front slope region of the response curve permits high sensitivity measurements while the back slope region is used for measurements at greater distances, where sensitivity, linearity, and accuracy requirements are less demanding. The "optical peak" region is used for optical inspection and comparison of surface conditions since at this position the output signal is more sensitive to light intensity variations than changes in displacement.

The distribution of fibers at the common (distal) end of the bundle is a major factor determining displacement range and slope sensitivity of the probe. For example, the greater displacement sensitivity is obtained with a staggered/random fiber array 6-8.

**Experimental Procedure**

**Sensor Setup**

Figure 2 indicates the experimental arrangement realised in the laboratory for determination of H2S gas concentration. Here light of a specific wavelength (sensitive to lead sulphide absorption) emanating from a high resolution monochromator is made to couple through suitable launching optics to the source arm of an optical fiber Y-guide fabricated in the laboratory. The light emanating from the common end of the Y-guide is scattered/ reflected from the brown stain on the lead acetate paper formed due to its exposure to H2S gas. The lead acetate paper is kept in close proximity of the common end of the Y-guide at a distance corresponding to the peak performance. The reflected light is captured by the detector arm of the fiber light guide and is made incident on a photo-detector and the corresponding output is displayed by a high sensitivity optical power meter. Figure 3 shows the photograph of the experimental bench set up realised in the Laboratory.

**Procedure for Preparation of Lead Acetate Papers**

Filter paper clippings of about 50mm x 50mm size were dipped in about 1 per cent lead acetate solution for about 5 minutes. These clippings were then removed
from the solution and dried at 110°C. A standard sulphide solution was prepared by dissolving 0.2438 gm of A.R. Sodium Sulphide in double distilled water in a volumetric flask and the volume of the solution was made to 100ml. This solution was diluted 100 times to obtain a 10ppm sulphide ion concentration.

In a series of cylindrical glass cuvettes of about 15ml capacity, 1, 2, 3, 4, 5, 6ml of 10ppm sulphide ion solution was taken and to each of the cuvettes 5ml of glacial acetic acid was added. Lead acetate papers were then placed on each cuvette and covered with a watch glass as to avoid the escape of hydrogen sulphide gas. The chemical reaction was allowed to take place for about 30 minutes to ensure complete release of the H2S gas. The gas thus produced reacts with lead acetate test papers producing a brown stain on them. The stained lead acetate test papers thus obtained were used for reflectance measurement.

**Results and Discussions**

Brown stains produced on the lead acetate papers exposed to varied concentrations of H2S were scanned by light of a specific wavelength through the fiber bundle and corresponding reflected output is given in Table 1. A linear correlation was observed between the H2S gas concentration and the reflected output intensity. A typical experimental curve obtained between these two parameters is given in Figure 4 which can also serve as the calibration chart for this sensor set up. Based on this experimental study, a compact instrument can be developed which will be very useful for on-line and remote monitoring of H2S gas concentration in industrial flow situations, such as in petrochemical and fertilizer industries.

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