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Abstract: This work discusses the results of a comparative study of two Coke's diagnostic methods. The results correspond respectively to tests using two radioisotopes with gamma scanning technique and radiotracers.

The first approach was presented in a study in which the structural characteristics of a laboratory constructed distillation column flow-rig) with 41 cm diameter have been investigated using a 241 Am isotope and tested using 99m Tc isotope. The Gamma scanning test consists in using the 241 Am as a gamma ray sealed source with the activity of 18, 5 GBq, associated to a NaI(Tl) detector which were applied to locate the column trays and coking.

The second approach by radiotracers consists of an injection of an appropriate quantity of a specific radiotracer at the inlet of the process and studying its presence in the column. The tracing is achieved by the measurement of the tracer concentration along the distillation column using four installed detectors.

After comparison and assessment between the two methods we conclude that the second approach with radiotracers achieved better results and therefore was more effective than the first one(the gamma scanning technique) for the detection of coking.

Keywords: coking, Gamma scanning, radiotracer, Americium 241, Technetium-99m

1. INTRODUCTION

Distillation columns are considered as one of the most important elements in oil refineries; The use of radioisotope technology in chemical, petrochemical and refinery processing vessels and columns has continuously been developed over the past 50 years to assist engineers in troubleshooting process problems, optimize production and minimize plant down time. Many advances in data acquisition and processing ability now allow critical information to be provided quickly to ensure the most efficient use of plant equipment. Radioactive tracers and radioisotope sealed sources are often commonly applied to the solution of problems on industrial plants to provide complementary information about the problem. [1]

Gamma ray scanning has become a popular diagnostic tool for troubleshooting of distillation columns. The unique and most powerful aspect of using this technique for the troubleshooting of distillation columns is that it is online and completely nondestructive. This technique is also a fast, efficient and cost–effective tool to better understand dynamic processes taking place in industrial columns and to examine inner details of a distillation column[2] [3].

The main beneficiaries of this technique are Petrochemical and chemical process industries because distillation columns are one of their main components and the efficiency of the industrial plant relies on the ability of these columns to work as they were projected [4].

This technique, often refer to as "column scanning", can be used for any type of columns such as tray type columns (one pass tray and double pass tray columns) and packed columns. The size of these columns in this technique varies in a large range. In order to scan these columns, no prepreparation is usually required and the scanning can be performed by accessing the platform while it is operating [5].

To detect the problems in packaging type distillation columns, scanning processes are made across the diameter of the columns. a gamma ray source and radiation detector is lowered simultaneously along

the opposite sides of a column .Then radiation intensity is recorded at different heights throughout the column. therefore, density profile of the distillation column is produced when the recorded radiation are plotted against the height. The profile is then correlated with the column internals to analyze the condition of the column [6][7].

The use of the radioactive tracers in industry started about fifty years ago, with the arrival on the market of diverse radioisotopes, and did not, since then, stop knowing a continuous extension. The success of the radioactive materials, to collect data which cannot be obtained by other techniques of investigation.

The importance of such studies is underlined by the fact that the competition to which are subjected the diverse chemical industries brings these to optimize their units' efficiency to make more competitive the made product. A better knowledge of the flow of fluids passing through devices contributes to reach this objective: we are led in certain cases to internal mechanical modifications of the devices which induce an improvement of their performances.

The implementation of radiotracers methods so allows to answer a certain number of questions concerning the characteristics of material transfer in the units without disrupting the operation. The main applications concern very diverse industrial domains such as the oil, the cement, the inorganic chemistry, the chlorine, etc..... [8].

Radiotracers play an important role in troubleshooting of processes in petrochemical industry

In this article we present the two major techniques used in petrochemicals, Gamma scanning and radiotracer for on-line troubleshooting of distillation columns, after the comparison between these two techniques radioisotopes for the detection of coking.

2. PRINCIPLES OF GAMMA RAY SCANNING FOR TROUBLESHOOTING IN DISTILLATION COLUMNS

At the time of a distillation column scanning (or of a similar, a small adequate sealed radioactive source and a detector are moved down simultaneously, with a small increment, on opposite sides along the lift of the column (Fig.1). A profile of relative density of the column's content is obtained. Analysing the obtained profile and comparing it to a mechanical scheme of the column, some conclusions can be deduced concerning the possible mechanical damages of the trays as well as some working conditions of the column, like obstructions, blockages, or other process's abnormalities



Fig1. Principle scheme of the Gamma-Ray scanning of a distillation column.

The quantity of gamma radiation absorbed or transmitted by the material placed between the radioactive source and the detector gives an indication of the nature and the real quantity of this material. The gamma radiation transmission through a material is given by the following equation (1):

$I = I_0 * exp^{-\mu\rho x}(1)$

Where:

I: Intensity of radiation transmitted through the material

 I_0 : Intensity of the incident radiation (without no interfering medium with the transmission of the radiation);

Coefficient of absorption of the measured material;

: Density of the material;

X: Thickness of the material (length of the course of the radiation through which it is transmitted).

The interaction of the radiation with the concerned medium results in variations of the intensity of the transmitted radiation beam. These variations can be analysed and linked to the properties of the medium inside a closed reservoir.

The gamma transmission equation through a medium describes an exponential attenuation which can be interpreted as function of the product's properties like the thickness and the density of the material (i.e. mass by surface unit) of the absorbing environment. These considerations, applied to the practical case of a column are translated by the following facts:

When radiation, issued from a radioactive source, pass through a medium containing a tray with an aired liquid, a good part of this radiation is absorbed and the radiation quantity reaching the detector is relatively small;

If a beam of radiation passes through a non aired liquid, the most part of this radiation is absorbed by the medium and the intensity is weak;

When the radiation beam passes through steam, in this case a small quantity of material is present to absorb the radiation. That means high intensities of radiation are transmitted to the detector [9].

To sum up, a scan using gamma radiation of a column can detect and localize of presence and formation of coking in packing bed.

3. GENERAL PRINCIPLE OF AN INDUSTRIAL APPLICATION OF RADIOTRACER

The principle of a tracer experiment is the one of any common method impulse-response (Figure 2): injection of a tracer in the entrance of a system and a recording of the concentration-time curve at the exit.



Fig2. General principle of radiotracer application in industry.

The operation consists in marking a slice of material, in the entrance of the device to be studied and to observe, in various characteristic points of this one, the response curve of the concentration of the tracer versus time, C(t).

Injection: the injection of tracers can be realized by various manners: very fast injection, injection with constant flow, injection in a very high-pressure circuit and under diverse physical forms: gas, liquid, powder. The injection will have to be the most brief possible so that the recorded response in the chosen points of measure can be considered as a Residence Time Distribution (RTD) of the marked phase. The tracer, packaged e.g. in a quartz bulb for gases, is placed in an injection device surrounded with a lead shielding.

Detection: Detectors allowing to measure through the walls of the installations the gamma rays, emitted by the tracer, are generally scintillation detectors type. The signals delivered by these probes are registered in a data acquisition system, monitored by a computer allowing the storage of the information of about ten sensors (or more) collected at high sampling rate. So, we can have, for every test, of the order of 5 000 information for each measuring channel. Later the data so stored are treated in batch mode by a computer in the laboratory[1].

4. MATERIAL AND METHODS

As shown in Fig .3, the experimental system consist of a distillation column in packing (flow-rig) (41cm diameter), column which has in Plexiglas's, a pump for circulating the water through the column, a gamma ray sealed source and a radiation detection system.

We used a Am-241 gamma ray sealed source housed in cylindrical container having collimator role with the activity of 500 mCi(18,5GBq). Also a 1×1 inch NaI (TI) scintillation detector was used in this experiment.



Fig3. Constructed laboratory on packed bed accompanied by the sealed source and detector

The source and detector were lowered simultaneously along the opposite sides of a column in steps of 1 Cm. The counter time interval of detection system was fixed to 10 seconds, coke is placed 30 cm at origin. The measurements have been recorded and repeated for three times. The average count value has been reported for each height, and then the relative density profiles for total count count were obtained, by plotting the graphs as: normalized counts vs. column height[10].

The second experimental in fig 4 by radiotracers consists of an injection of an appropriate quantity of a specific radiotracer at the inlet of the process and studying its presence in the column.

We used a using Tc -99m tracer no sealed source .Also a .Also aThe tracing is achieved by the measurement of the tracer concentration along the distillation column using four installed detectors 1×1 inch NaI (TI) expecting that a channeling effect would be observed in flow circulation inside the column caused by the presence of coke



Fig4. Constructed on packed columns model accompanied by experimental setup for radiotracer

5. RESULTS AND DISCUSSION

5.1. Gamma Scanning

A scanning grid was therefore performed on a packed bed structured to detect the presence and formation of carbon in the bed.

The results obtained are shown graphically (Figure-5.a and Figure-5.b), they are also indicated in the figures with the trays' positions and the transmitted radiation, identified from a mechanical point of the column. All distances in the figures refers to an origin point located above the tray 1.

According to the use of two scan profiles obtained with and without coke, under the same fluid circulating conditions, show that it is not possible to locate the coke position inside the column packing.

The regular-attenuation peaks coincide with the positions of the tray 1 and 2 for the two cases; we conclude that the plates were in place at the time of scan.

The analyzes of the two scans used in our experiment did not allows us to deduct the position of the coke in the lined bed.



Fig5.a. Scan profiles obtained on packed bed an absence of coke

Fig5.b. *Scan profiles obtained on packed columns (bed) in the presence of coke*

5.2. Radiotracer

Injecting a compatible radiotracer into an injection point in input an appropriate container and monitoring its passage through the container allows the delivery of tracer to be measured.

In this work we have scintillation detectors sensitive to gamma radiation. (A total of five detectors were mounted around the column. One detector was used to monitor the inlet of the tower for timing. The remaining 4 detectors (1×1 inch NaI (TI)) were mounted in front of the tower: two were placed in front of the top of the tower and the other two are mounted in front of packed bed bottom. For simplicity, the detectors' locations will be referred to as V1, V2, V3 and V4 (Figure-6).



Fig6. Simple schema Column with 4 detectors and the position of coking for the study

(The data collected, from detectors placed to monitor flow distribution, were used to calculate Vi/Vj ratios to get an estimate of the flow. The tables below show the relationship of the response of the sensors in different ways with, and without, coke.

Ways	With coke (cts)	Without coke (cts)	Ways	With coke	Without coke
Voie 1	537288	470342	V2/V4	2,5	1,8
Voie 2	146504	136259	V3/V1	1,1	0,6
Voie 3	597237	298524	V2/V3	0,25	0,46
Voie 4	71505	76178	V4/V1	0,13	0,16

Table-1-a & b. Comparaison of total activity "seen" by each detector

As the used distributor was a linear one, it is supposed that the liquid flow is uniform along a longitudinal cross section of the upper part of the column. However, value greater or less than 1 for the V2/V4 and V3/V4 ratios, without coke, indicate the contrary. But increasing values from 1.7886 (without coke) to 2.0488 (with the presence of coke) in the case of the V2/V4 ratio refers to obstruction of the channel 4, which can be explained by the presence of the coke on top of channel 4. In the same way, in the case of the V3/V1 ratio, it is observed an increase resulting from a high concentration of the tracer due to a channelling effect when we are in presence of coke.)



Fig7. The processed output curves of all detectors in the absence of coking





В







Fig8. The processed output curves of all detectors in the presence of coking

The channeling effect in the case of our experiment has been estimated to 20%



Fig9. Comparaison of total activity "seen" by each detector

6. CONCLUSION

This work presents the experimental work for inspection of the structural characteristics of a laboratory distillation column, using both techniques gamma scanning and radiotracer for detecting coke.

The principles and scope of the application of radioisotopes and ionizing radiation in industry are reviewed as an introduction to nuclear methods of control in refining. The potential of the radiotracer technique is emphasized in of reported investigations.

Although column scanning using sealed sources remains a valuable technique for troubleshooting in many cases, it has limited application when it comes to coking phenomenon detection in packed bed columns

According to the results, the experimental study carried out shows that radiotracer method is more effective than the gamma scanning technique for the detection of coking in packings.

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