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Measurements of liquid phase residence time distributions in a pilot-scale continuous leaching reactor using radiotracer technique

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HIGHLIGHTS

- Radiotracer technique was applied for evaluation of design of a pilot-scale continuous leaching reactor.
- Mean residence time and dead volume were estimated. Dead volume was found to be ranging from 4% to 15% at different operating conditions.
- Tank-in-series model was used to simulate the measured RTD data and was found suitable to describe the flow in the reactor.
- No flow abnormality was found and the reactor behaved as a well-mixed system. The design of the reactor was validated.

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ABSTRACT

An alkaline based continuous leaching process is commonly used for extraction of uranium from uranium ore. The reactor in which the leaching process is carried out is called a continuous leaching reactor (CLR) and is expected to behave as a continuously stirred tank reactor (CSTR) for the liquid phase. A pilot-scale CLR used in a Technology Demonstration Pilot Plant (TDPP) was designed, installed and operated; and thus needed to be tested for its hydrodynamic behavior. A radiotracer investigation was carried out in the CLR for measurement of residence time distribution (RTD) of liquid phase with specific objectives to characterize the flow behavior of the reactor and validate its design. Bromine-82 as ammonium bromide was used as a radiotracer and about 40–60 MBq activity was used in each run. The measured RTD curves were treated and mean residence times were determined and simulated using a tanks-in-series model. The result of simulation indicated no flow abnormality and the reactor behaved as an ideal CSTR for the range of the operating conditions used in the investigation.

1. Introduction

Uranium as sodium diuranate ($Na_2U_2O_7$) is used as a fuel in Indian nuclear reactors for power production (Gupta and Sarangi, 2005; Gupta, 2010). The production of $Na_2U_2O_7$ involves a series of unit operations. Uranium ore (0.03–0.04%) as received from the mine is subjected to dry crushing in a Jaw Crusher followed by a Roll Crusher. The crushed ore is ground further to a particle size of 100 μm in a closed loop wet grinding mill. The ground ore is separated from liquid and is subjected to a leaching process. The leaching is the most important process in uranium extraction and is carried out either in an alkaline or acidic medium. In an alkaline based leaching process, the ground ore is mixed with Na_2CO_3 and $NaHCO_3$ or with recycled leach filtrate obtained from the previous cycle. Water is also added to adjust the slurry density approximately to 50%. The carbonate–bicarbonate balancing in slurry is achieved by addition of sodium carbonate or injection of carbon dioxide gas as per the process requirement. The prepared feed is pumped to a pressurized leaching vessel (autoclave) at a predetermined flow rate where it is stirred and maintained at a temperature of 125–135° C and at a pressure of 6–8 kg/cm^2. The leached slurry is pumped out from the reactor and fed to a heat exchanger prior to its depressurization while maintaining the steady state flow condition in the reactor. In the heat exchanger, the leached slurry exchanges the heat with the incoming cold feed slurry where its temperature is reduced to 40 °C. After removal of the heat, the leached slurry is depressurized and transferred to a filtration unit. The uranium present in the leach filtrate is recovered in the solid form by precipitating out as sodium diuranate (SDU) commonly known as yellow cake. The precipitated SDU is filtered, washed and dried and stored as a product; and

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subsequently used to fabricate fuel bundles/rods (Gupta and Sarangi, 2005; Gupta, 2010).

A continuous leaching reactor (CLR) based on alkaline leaching of uranium was designed, installed and operated for the leaching process at a Technology Demonstration Pilot Plant (TDPP) in India. The efficiency of the leaching process depends upon the effectiveness of the leaching and the design/performance of the reactor used for the leaching. The leaching process had already been studied and optimized. However, it was felt necessary to evaluate the hydrodynamic behavior of the reactor and the connected heat exchanger. Residence time distribution (RTD) analysis is very useful to access the design of the continuously operating chemical process equipment and optimize their operating parameters (Levenspiel, 1972). Radiotracer techniques are commonly used for measurement of RTD of flowing process material in pilot as well as full-scale industrial systems and determine flow parameters such as mean residence time (MRT), bypassing, extent of dead volume present and degree of mixing (Charlton, 1986; IAEA, 1990; Thyn et al., 2000; Pant et al., 2001a, 2001b, 2009a, 2009b, 2012; Pant and Yelgoankar, 2002; Samantray et al., 2014). The results of RTD analysis obtained in pilot-scale systems are utilized to scale up the process. This paper discusses radiotracer investigations carried out for measurements and analysis of liquid phase RTD in pilot-scale CLR setup at the uranium processing Technology Demonstration Pilot Plant (TDPP) for uranium processing in India.

2. Continuous leaching reactor

The continuous leaching reactor is a horizontal autoclave with three interconnected compartments. The volume of each compartment is 260, 175 and 150 l, respectively. Each compartment has an agitator and the feed slurry overflows from one compartment to the next. Outlets for drainage of water or process material, whenever required, are provided at the bottom of the second and third compartments of the reactor. The schematic diagram of the reactor is shown in Fig. 1. The reactor is made of inconel alloy to avoid corrosion and designed to operate at a pressure and temperature of 15 kg/cm^2 and 200 °C, respectively. The feed rate to the reactor is monitored with an on-line magnetic flow meter. The liquid levels in the first two compartments are determined by the compartment overflow weir heights, whereas the level in the last compartment is monitored by the differential pressure type level transmitter. Oxygen (O_2) and carbon dioxide (CO_2) gases are introduced intermittently to the reactor, which do not cause any appreciable increase in the slurry volume. No foaming is expected due to the CO_2 and O_2 purging. The leaching is carried out at a temperature between 125 and 135 °C and at a pressure of 5 kg/cm^2. All the three compartments of the reactor are provided with separate steam jackets having dedicated steam inlet, condensate outlet, safety relief valve and temperature transmitters for better temperature control during the course of leaching. Maximum steam consumption is expected in the first compartment and therefore a control valve is provided in the steam line. The second and third compartments are fed with steam at 3 kg/cm^2(g). The slurry temperature in all the three compartments is monitored using the dedicated temperature transmitter mounted in each compartment. Cooling of the reactor is achieved by circulating cooling water. The pressurized leached slurry from the reactor is fed into a heat recovery system to bring down the temperature to about 40 °C prior to its depressurization. A spiral heat exchanger connected in series with the reactor is used for cooling the leached slurry before further processing.

3. Radiotracer investigation

The schematic diagram of the CLR and experimental setup is shown in Fig. 1. The investigations were carried out with water as flowing phase and at ambient conditions. The outlets at the bottom of the second and third compartments were used for out flow of water during the experiments. Eight radiotracer tests were carried out for RTD measurement of liquid phase in the CLR at selected operating and process conditions and are given in Table 1. The first five runs were carried out in the first compartment of the CLR, whereas the remaining three tests were carried out with both the compartments connected in series. Bromine-82 as ammonium bromide was used as a tracer for tracing the aqueous phase. The tracer was instantaneously injected into the inlet feed line of the reactor using a specially fabricated injection system and monitored at the bottom outlets as shown in Fig. 1 (Samantray et al., 2014). The bottom drainage outlets were used for monitoring of the radiotracer with intention to investigate the mixing characteristics of the individual compartments. About 40 MBq activity was used for RTD measurements in the first compartment (Run 1–Run 5) whereas about 60 MBq activity was used for measurements in the case of the first and second compartments interconnected in series (Run 6–Run 8). The tracer was monitored using collimated scintillation detectors connected to a computer controlled data acquisition system preset to acquire data at an interval of 20 s.

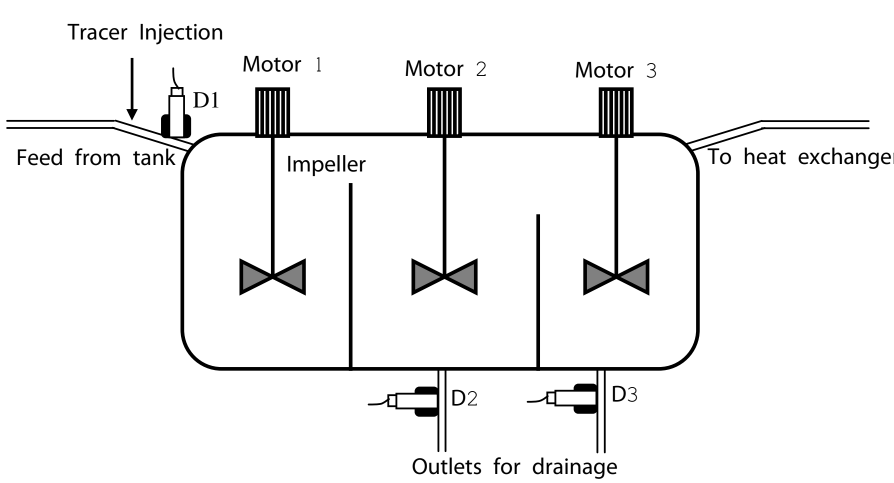

Fig. 1. Schematic diagram of continuous leaching reactor and experimental setup.