IChemE
www.icheme.org/journals
doi: 10.1205/cherd.03414

0263–8762/05/$30.00+0.00

Trans IChemE, Part A, April 2005
Chemical Engineering Research and Design, 83(A4): 344–351

CONSIDERATIONS FOR THE SELECTION AND DESIGN OF SAMPLING SYSTEMS FOR HETEROGENEOUS PROCESSES IN A STIRRED TANK REACTOR

A. DIEZ-LAZARO, M. L. HITCHMAN and D. LITTLEJOHN*

Department of Pure and Applied Chemistry/CPACT, University of Strathclyde, Glasgow, UK

On-line process analysis requires material to be passed through a sampling line to an analyser for measurement. With batch processes, this is often achieved using a fast sampling loop. In this work, a review of sampling systems for stirred vessels is presented. The aim is to outline the main aspects to consider when selecting and designing an effective sampling device for a stirred tank reactor. The importance of factors such as probe location, orientation of the probe and sampling velocity is highlighted. The main conclusion is that the sample probe should be positioned horizontally at the stirrer plane, between the stirrer blade and the reactor wall, in order to achieve optimized sampling from heterogeneous systems. It is also preferable to use a pump able to provide a flow rate at least equal to that required for isokinetic sampling conditions in the fast loop.

Keywords: sampling; batch reactors; heterogeneous proceses; on-line analysis.

INTRODUCTION

On-line process analysis is often an integral part of the control systems used in the chemicals, pharmaceutical, biotechnology, food and other industries. In on-line analysis, process material is automatically removed from the reactor and transferred to an instrument to determine some chemical or physical property of the sample. The material is then either recycled back into the process or removed to waste. Proper operation of the analyser is clearly important if useful data are to be obtained for control of the process. However, the efficacy of the analysis depends equally on the sampled material being an appropriate indicator of the status of the reactor (Podkulski, 1997). The main issues to consider when designing a sampling system for on-line process analysis are the representativeness of the sample, compatability of the sample with the analyser, timeliness and reliability of sampling, safety and environmental concerns, and the overall cost of the sampling system (Carr-Brion and Clarke, 1996; McLennan and Kowalski, 1995).

Typically, a sampling system for a gas or liquid consists of a sampling point or probe, pipelines, filters, pressure regulators, valves, pumps, flow meters, condensers or vaporizers, pressure gauges and other components. Selection of these parts, the design of the components, and their adaptation to the system, have to be carefully considered. Poor sampling will lead to erroneous results even with the most reliable analysers.

For batch reactors, sampling would seem to be quite simple as, in theory, material from any point inside the vessel possesses the same properties if the mixture is well stirred. However, in real industrial processes this is not often the case and further aspects must be considered in order to get representative information, especially for heterogeneous processes (Carr-Brion and Clarke, 1996). The effect of the flow-patterns inside the reactor, the stirring speed, the velocity in the sampling line, and the shape, size and angle of the sampling probe are some of the factors that must be carefully considered when designing sampling systems for chemical reactors. The importance of these parameters is considered in this review, with an emphasis on the sampling of heterogeneous systems, such as agitated particles in a liquid medium.

GENERAL CONCEPTS OF SAMPLING

There are many measurement situations where a fast sampling loop and pump are used to transport the material from a sampling probe in the reactor to a conditioning system which adjusts the sample to the temperature, flow rate, pressure, and so on, that are compatible with the analyser. A 'slow loop' is then often used to deliver, at a lower flow rate, a secondary sample stream to the analyser (Hassell and Bowman, 1998). The shape, orientation and dimensions of the sampling probe affect the quality of the extracted sample and must be designed carefully. Two main factors affect particle collection in heterogeneous

---

*Correspondence to: Professor D. Littlejohn, Department of Pure and Applied Chemistry/CPACT, University of Strathclyde, Glasgow, G1 1XL. E-mail: d.littlejohn@strath.ac.uk

processes: particle inertia and the particle bouncing effect (MacTaggart *et al.*, 1993). Particle inertia effects are observed when the trajectory of the heavier particles deviates from the lighter fluid streamlines. The magnitude of this effect depends upon factors such as the size and density of the particles, the viscosity of the solvent and the local velocity of the particles at the sampling point (Carr-Brion and Clarke, 1996; Nasr-El-Din *et al.*, 1996). The particle inertia is represented by the dimensionless Stokes number, *k*, which is given by the following expression, for solid particles immersed in a fluid (Kuriyama *et al.*, 1996):

$$k = \frac{\rho_{\mathrm{p}} d_{\mathrm{p}}^2 U_0}{18\mu_{\mathrm{L}}(\phi/2)} \tag{1}$$

where

d_{p} = particle size diameter, m
ρ_{p} = particle density, kg m^{-3}
U_0 = local velocity, m s^{-1}
ϕ = sample tube inside diameter, m
μ_{L} = liquid viscosity, kg m^{-1} s^{-1}

If a similar expression for *k* is to be developed to quantify the inertia for two liquid phases, then d_{p} in equation (1) would have to include the average size of the droplets of the heavier phase. However, this average size is sometimes difficult to predict and is highly dependent on the stirring speed and the viscosity of the fluids, and may even change with time.

Particles giving a high value of *k* do not follow the fluid streamlines, but tend to travel in straight lines resulting in serious sampling errors as the particle and fluid velocities are not collinear. The particle bouncing effect occurs when a particle moving in the fluid strikes a surface and loses some of its momentum. If this happens near a tube opening, the reduction in momentum can be enough to allow particles to be extracted when they otherwise would not have been.

The flow streamlines in stirred tank reactors must be considered, to deal with the problem of particle inertia (MacTaggart *et al.*, 1993; Nasr-El-Din *et al.*, 1996; Nasr-El-Din, 1987), and the dimensions and shape of the probe must be carefully designed to minimize the particle bouncing effect at the desired sampling point (MacTaggart *et al.*, 1993). In a fast sampling system, the loop length must be as short as possible and the loop velocity as fast as practicable in order to reduce any associated lag time and improve timeliness (Carr-Brion and Clarke, 1996). Some authors (Richardson and Peacock, 1994) consider the effects of the reaction continuing inside the sampling system to be negligible when the dead time, i.e., the time to traverse the distance from the sample point to the analyser, is less than 30 s. The applicability of this guideline depends on the kinetics of the reaction.

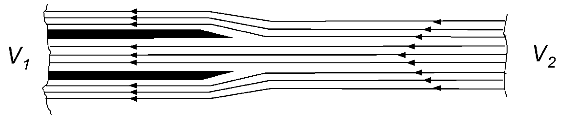
The sampling velocity also affects the performance of the sampling system. This effect is more important in heterogeneous mixtures of solids or liquids in gases than in mixtures of immiscible liquids. However, several studies have shown how the wrong choice of sampling velocity leads to the collection of samples that are unrepresentative of a process involving suspended solids in liquids (MacTaggart *et al.*, 1993; Nasr-El-Din *et al.*, 1984, 1996, Rushton, 1965; Barresi *et al.*, 1993, Barresi *et al.*, 1994a, 1994b; Kuzmanic *et al.*, 1992). Isokinetic sampling is achieved when the sampling velocity equals the local system velocity at the point of sampling. This type of sampling must be used in order to ensure representative sampling from heterogeneous processes in many stirred tank configurations (Carr-Brion and Clarke, 1996; Liptak, 1994; Baldi *et al.*, 1981). If the sampling velocity is higher than the isokinetic value, super-isokinetic sampling is achieved and an excessive amount of lighter particles enters the probe. However, under sub-isokinetic sampling conditions, portions of the light phase stream are deflected; the lighter particles follow the deflected stream and are not collected, while the heavier particles continue into the probe due to their inertia (Liptak, 1994). In super- or sub-isokinetic conditions, an unrepresentative sample of the process is usually obtained (Carr-Brion and Clarke, 1996). These three types of sampling conditions are illustrated in Figure 1.

SAMPLING FROM STIRRED VESSELS

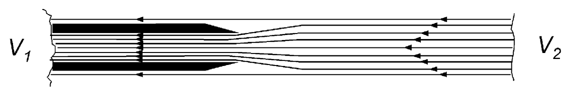
In the area of sampling from stirred vessels, not much progress has been made since Rushton performed one of the first studies (Rushton, 1965). He studied the effect of the position and size of the withdrawal pipe opening on the ratio of two phases sampled from a stirred tank. Experiments were performed using glass beads suspended in water in a baffled tank provided with turbine-type mixing impellers (the so called Rushton turbine). Rushton proposed equations describing the local velocity at the stirrer plane as a function of the radial distance to the impeller blade. He also defined equations describing the effect of the sampling velocity on the quality of the sample collected. More attention will be paid to these findings later in this article.

Sharma and Das (1980), considered Rushton's work and proposed the stirrer plane as the best place to withdraw samples in stirred vessels. They analysed the data obtained when sampling from a point at the stirrer plane and at different sampling velocities. They also defined a model that relates the quality of the sample with the sampling velocity.

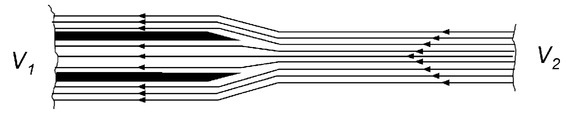
At the start of the 1980s, Baldi *et al.* (1981), defined some examples of concentration profiles in stirred vessels



$V_1 = V_2$ (Isokinetic sampling)



$V_1 > V_2$ (Non-isokinetic sampling)



$V_1 < V_2$ (Non-isokinetic sampling)

Figure 1. Types of sampling depending on the sampling velocity.