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Ultrasonic measurement of solids concentration in an autoclave reactor at high temperature

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Abstract

An ultrasonic technique was developed to measure the slurry concentration in an autoclave reactor. Preliminary measurements were conducted on slurries consisting of molten FT-200 wax, glass beads, and nitrogen bubbles at a typical Fischer–Tropsch (FT) synthesis temperature of 265 °C. The data show that the velocity and attenuation of the sound are well-defined functions of the solid and gas concentrations in the molten FT-200 wax. The results suggest possibilities for directly measuring solids concentration during operation of a three-phase slurry reactor under the reaction temperature and with molten FT-200 wax. © 1997 Elsevier Science S.A.

Keywords: Ultrasonic; Solids concentration; Autoclave reactor

1. Introduction

Fischer–Tropsch (FT) synthesis represents an important route to convert coal-derived synthesis gas into premium-quality fuels, such as light hydrocarbon, gasoline, or diesel fuels depending on the catalyst employed, the reaction temperature, and the process employed. The slurry phase Fischer–Tropsch process is considered a potentially economic method to convert coal-derived synthesis gas into liquid fuels, largely due to its relatively simple reactor design, improved thermal efficiency, and ability to process CO-rich synthesis gas. The application of the three-phase slurry reactor system for coal liquefaction processing and chemical industries has recently received considerable attention. To design and efficiently operate a three-phase slurry reactor, the degree of dispersion of the solid (catalyst) in the reactor must be understood and controlled. The solids distribution within the reactor greatly affects its performance. Because it is crucial to understand the influence of various reactions and reactor configurations on the solids concentration profile, measurement of solids concentration must be made under reaction conditions, such as high temperature, pressure and with the presence of reaction liquid medium.

Several 'on-line' techniques exist to monitor the concentration of solids in a three-phase slurry. Most of these methods are optically based techniques which use reflected, scattered, or absorbed light to measure the concentration of solids. These methods are sensitive to the opacity of the slurry phase [1]. The direct sampling of solid particles from a slurry reactor suffers from either a need to perturb the reaction system or a difficulty of measurement because of high pressure and temperature [2]. The static pressure method also suffers some difficulties when the solid and liquid densities are close [3].

Ultrasonic measurement is an established technique for many industrial applications, for example medical imaging, materials testing, flow detections, and level measurements [4–6]. The ultrasonic technique has advantages over many existing methods because it is a non-invasive and non-destructive measurement in systems which are concentrated, optically opaque, and electrically non-conducting [6]. However, the main disadvantage of this technique is that there are no commercially available ultrasonic instruments which are designed to characterize the concentration of solids. Therefore, researchers have to develop their instruments and data interpretation. The utilization of ultrasonic techniques for slurry characterizations has received considerable attention recently [7–14]. Some ultrasonic techniques are based on the principle of scattered acoustic pulses. The process is similar to sonar, in which bursts of acoustic energy are emitted into a liquid and reflections from discontinuities (solid and bubbles) are detected and measured. Abts and Dahi [7] have

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applied the ultrasonic technique to determine the concentration of oil droplets in an oil recovery system by detecting the forward-scattering of ultrasonic energy from oil droplets in the oil recovery system. Foote [8] has reported that a particulate in a fluid can be identified by sending an ultrasonic pulse across the fluid, measuring the amplitude of the portion of the pulse scattered from a particulate at a preselected angle. The relative size of the particulate is determined from the magnitude of the scattered signal. An on-line ultrasonic particle monitoring for brewing operations has been reported by Behrman and Larson [9]. These ultrasonic techniques are based on the scattering of ultrasonic pulses. The use of the ultrasonic Doppler technique to characterize the local bubble rise velocity in a bubble column reactor has been investigated by Hilgert and Hofmann [10]. Bonnet and Tavlarides [11] have applied an ultrasonic transmission technique to determine the dispersed-phase hold-up of liquid–liquid dispersions by measuring the velocity of ultrasound in suspensions and emulsions. Tsouris et al. [12] have used an ultrasonic technique for real-time hold-up monitoring for the control of extraction columns. Furthermore, Tsouris et al. [13] have applied a pulse-echo ultrasonic probe for local volume fraction measurements in liquid–liquid dispersions. Warsito et al. [14] have conducted simultaneous measurement of gas and solid hold-ups in a bubble column using ultrasonic methods. Recently, a method involving the measurement of ultrasound transmission has been reported in a slurry-phase stirred-tank reactor which offers the possibility of using the ultrasonic technique for the measurement of solids concentration in a three-phase slurry reactor [15]. The ultrasonic transmission uses measurements of the velocity and attenuation of the sound wave which travels directly through the slurry sample. When an acoustic wave strikes the boundary between two different media (liquid and solid) and the acoustic impedances of the two media are different, some acoustic energy will be reflected, some absorbed, and some will be transmitted. The reflected wave travels back through the incident medium (liquid) at the same velocity. The transmitted wave continues to move through the new medium (solid) at the sound velocity of the new medium. When the velocity of sound in a liquid (1324 m s^{-1} at 25 °C for kerosene) is significantly different from that in a solid (5968 m s^{-1} at 25 °C for fused silica), a time shift (a velocity change) in the sound wave can be detected when solid particles are present relative to that for the pure liquid. The amplitude of the sound wave is also reduced when a solid particle is present since the wave is partially scattered and absorbed. Therefore, a change in amplitude of the sound wave can also be detected when solid particles are present relative to that for the pure liquid. Okamura et al. [15] used a continuous stirred-tank reactor to correlate the concentration of solids to the relative time shift $((t_a - t_b)/t_o)$. The arbitrary first distinct zero crossing time in liquid and in solid–liquid are defined as t_a and t_b, respectively. The travel time between the transmitter and receiver in the liquid is defined as t_o. Thus, the concentration of solids in a slurry reactor can be measured by sending an ultrasonic pulse across the slurry and measuring the amplitude and time shift of that portion of the transmitted pulse received at the opposite side of the reactor. Then, comparing the value with those for known concentration, the concentration of solids is determined from the measured signal.

Recent studies reported from our laboratories for the water/nitrogen/glass beads system at ambient conditions indicated that both the amplitude and the fractional change of transit time were affected by the variation of solids concentration (up to 35 wt.%) under a constant nitrogen flow in the reactor [16,17]. This study examines the possibility of utilizing the ultrasonic technique for slurries concentration measurements in an autoclave reactor under the conditions prevailing in the Fischer–Tropsch synthesis — temperature of 265 °C, glass beads, nitrogen bubbles and molten FT-200 wax.

2. Experimental details

Fig. 1 shows a schematic representation of the 2.5 l autoclave reactor in which the ultrasonic investigation was conducted. The reactor vessel was a flat bottom stainless steel cylinder, 10 cm in diameter and 30 cm in height, fitted with four equally spaced, vertical baffles, each 1/8 of the diameter of the vessel in width. The slurry was mechanically agitated by a motor-driven stirrer located 4 cm from the bottom of the vessel. A standard three-blade stirrer (4 cm in diameter) with a constant stirring speed of 600 rev min^{-1} was used in this study and was slightly off the center of the reactor to avoid any interference with the ultrasonic signals, which were transmitted at 7 cm above the bottom of the reactor along the center of the reactor. Nitrogen bubbles were introduced

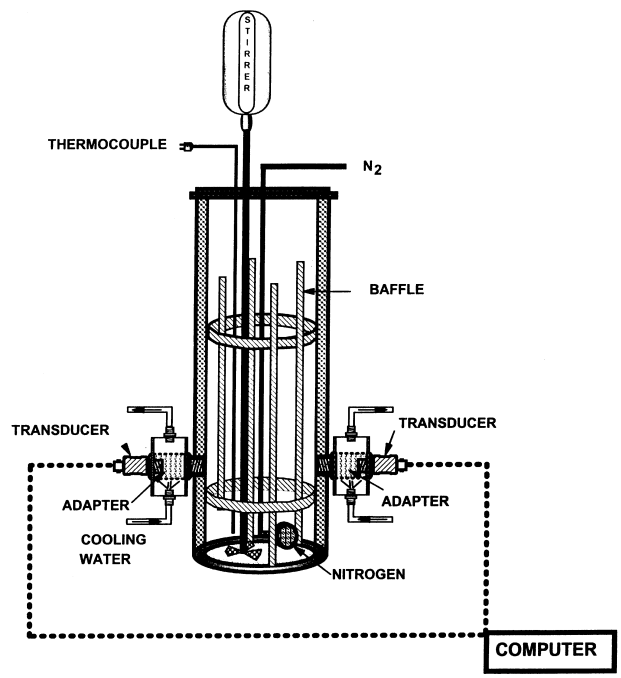

Fig. 1. Schematic diagram of the autoclave reactor.

through a sintered frit at the bottom of the reactor. The nitrogen flow was controlled electronically to a maximum of 400 ml min^{-1} through a mass-flow controller. Glass beads from Cataphote, Inc., (10–37 μm in diameter with density of 2.46 g cm^{-3}) were used as the solid in the slurry. The concentration of solids (solid weight/total weight) was varied from 1 wt.% to 25 wt.% for each nitrogen flow in the reactor. Molten FT-200 wax (Vestowax), with an average molecular weight of 600, at a temperature of 265 °C was used as the liquid medium. The density and viscosity of the FT-200 wax at 265 °C are 675 kg m^{-3} and 1.9 mPa s, respectively [20]. Experiments were conducted at 0.1 MPa and 265 ± 0.5 °C in this study. Ultrasonic measurements were taken by using a computer-based TestPro system, manufactured by Infometrics Inc., Silver Spring, MD, USA. The ultrasonic unit was composed of a pulser/receiver board and an A/D and D/A board, both of which were controlled by a COMPAQ computer. The software signals the pulser/receiver board to deliver a voltage of a certain amplitude, duration, and frequency to the transducer which generates the ultrasonic pulse by energizing the piezo-electric crystal causing it to vibrate at a certain frequency in the ultrasound region. The transducer then emits an ultrasonic pulse which travels through the slurry in the reactor, and the receiver at the opposite side of the reactor receives the pulse. This information was then analyzed by the TestPro software. The software can specify different types of pulse shape. In this study, bipolar-positive was chosen for the pulse shape. The software can also control the gate length of the pulse by setting the initial and length of the gate. The length of the gate was set at 5.12 μs. The output pulse can be programmed up to eight cycles, emitted every 400 ns, so that the transducer could ring down and the echoes die between pulses. A typical pulse has an amplitude between 1×10^{-6} and 1×10^{-3} V, pulse duration of 2.5 MHz and a sampling frequency of 10 MHz. The data were obtained with longitudinal waves at a frequency of 2.5 MHz using lithium niobate transducers (transmitter/receiver). Both the transmitter and receiver were mounted inside a metal adapter which was screwed into a fitting inside the reactor wall as shown in Fig. 1. The cooling water was circulated through a cooling collar to the adapter to prevent the transmitter/receiver from being heated.

3. Results and discussion

Fig. 2(a) shows a typical ultrasonic wave propagating in FT-200 wax at 265 °C with a constant stirring in the set-up described in this study. Since the received signal is undistorted, the first distinct zero crossing time is chosen as the transit time. Here, t_a is the arbitrary first distinct zero crossing time in liquid ($t_a \approx 153.2$ μs). The travel time between the transmitter and receiver in the liquid is defined as t_o ($t_o \approx 152.84$ μs).

Fig. 3 illustrates the change in the amplitude ratio of the transmitted ultrasonic signals A/A_o in the reactor as a function

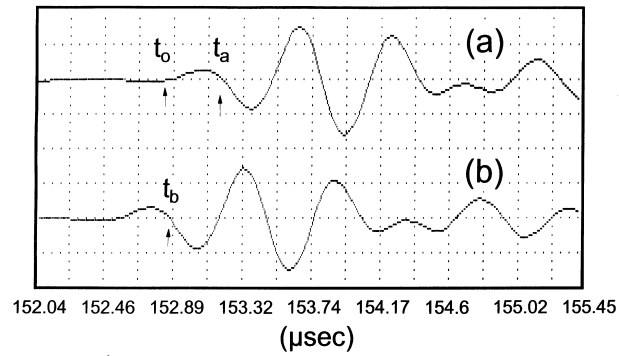

Fig. 2. (A) Typical received signal in the autoclave reactor with FT-200 wax only. (b) Effect of solids on the ultrasonic signal.

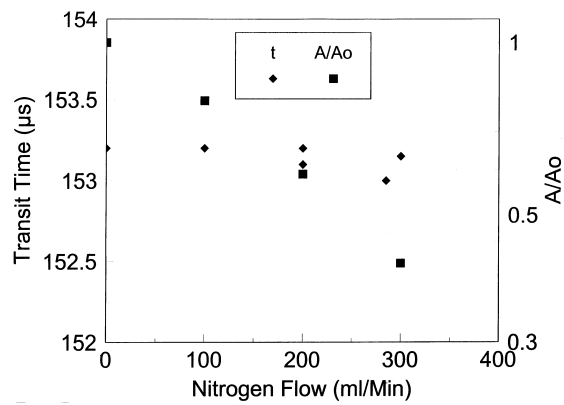

Fig. 3. Amplitude ratio and transit time as a function of nitrogen flow in FT-200 wax at 265 °C.

of nitrogen flow in FT-200 wax at 265 °C. A and A_o are the amplitudes of the transmitted signals with and without the presence of nitrogen, respectively. The transit time and amplitude measurements in this study are average values over five to seven different measurements. The standard deviation on the collected data was around 0.05% of the average value. Fig. 3 also suggests that the amplitude ratio be approximately an inverse exponential function of the nitrogen flow. As described in the introduction, the impedances of the two media will determine the transmission of the wave from one medium to another and the amount of reflection of sound at the boundary between the two media. If the impedances of two media are widely separated, e.g. nitrogen and FT-200 wax, then most of the energy is reflected back in the first medium (FT-200 wax) with little transmission into the second medium (nitrogen). It can be assumed that the ultrasonic pulse cannot penetrate through much of the nitrogen/FT-200 wax interface at the current experimental frequency due to the acoustic impedance mismatch of this combination. Therefore, the amount of attenuation of the ultrasound beam by nitrogen bubbles is proportional to the gas volume fraction but also can be dependent on bubble size present in the path of the ultrasound, especially when the ultrasound wave is near the resonance frequency. The attenuation is greatest at frequencies near the resonant frequency [6]. For this study, the frequency utilized is not near the bubble resonance frequency. Therefore, the data collected is not affected by the

resonance effect. We also measured the bubble sizes under the same experimental conditions through a dual hotwire anemometer under separate experiments. The majority of the bubble diameters was found to be about 5 mm in the path between the transducer and the receiver within the range of flow studied. The number of bubbles increased as the nitrogen flow rate increased. The decrease of A/A_o as the nitrogen flow increased appears to be related to the bubbles. Chang et al. [18] measured void fractions up to 20% in bubbly air–water two-phase flow using an ultrasonic transmission technique. Their results showed that the transmitted ultrasonic signal could be approximated by the exponential relationship:

$$A/A_o = \exp[-f(d_b)\epsilon] \quad (1)$$

where ϵ is the void fraction and $f(d_b)$ is a function dependent on the Sauter mean diameter. This correlation shows that the A/A_o ratio has an exponential relationship with both the void fraction and with a function dependent on the bubble diameter. The effect of air bubble diameter on the A/A_o ratio was found to be significant, with A/A_o decreasing with increasing bubble size. Bensler et al. [19] also measured the interfacial area in bubbly flows in air–water systems by means of an ultrasonic technique. Their observations showed that the transmitted ultrasonic signal could also be expressed by an exponential relationship:

$$A/A_o = \exp[\Gamma x/8\, S(kd_b/2)] = \exp[\Gamma x/8\, S(k3\epsilon/\Gamma)] \quad (2)$$

where Γ is the volumetric interfacial area, x is the travel distance in the path, S is the scattering coefficient, k is the wavenumber of the ultrasonic waves which surrounds the bubble, ϵ is the gas hold-up and d_b is the Sauter mean bubble diameter. Eq. (2) shows that the A/A_o ratio has an exponential relationship with the interfacial area and the scattering cross-section, which is a function of both the bubble radius, gas hold-up and the wavenumber of the ultrasonic wave surrounding the bubble. Our observations of A/A_o in the nitrogen/FT-200 wax system are in qualitative agreement with those reported by Chang et al. [18] and Bensler et al. [19]. The decreasing A/A_o ratio as the nitrogen flow increased in Fig. 3 may be attributed to a combination of the void fraction, bubble size, the number of bubbles, and the scattering cross-section. Fig. 3 also shows the effect of the nitrogen flow on the transit time, t, which was approximately 153.2 μs at all nitrogen flows. Apparently the transit time was unaffected by the nitrogen flow under the current experimental conditions, because what we measured was the signal not transmitted through the nitrogen. Thus, the measured transit time should not be affected by the nitrogen flow. The results indicate that only the amplitude and not the transit time of the ultrasonic signal are affected by the nitrogen flow rate in the reactor under the current experimental conditions. Chang et al. [18] also reported that the amplitude of the transmitted sound pulses depends significantly on the number of bubbles; however, the transit time does not change with the void fraction. Our results obtained from the nitrogen/FT-200 wax system are similar to those of Chang et al. [18] for the air/water system. It is known that the temperature of wax greatly affects the acoustic velocity. Therefore, an error will occur in determining the velocity due to the variation in temperature. To overcome this complication, the temperature in the reactor was controlled at 265 ± 0.5 °C in this study.

Fig. 2(b) shows a typical ultrasonic signal as affected by 3 wt.% of solids. Clearly, the presence of the solids caused a substantial decrease in the transit time. The first distinct zero crossing time, t_a, which is 153.2 μs in Fig. 2(a), is shifted to 152.88 μs in Fig. 2(b), after the addition of 3 wt.% of solids. Fig. 4 further shows the effect of solids concentration on transit time and the amplitude ratio of the transmitted ultrasonic signal A/A_o (A and A_o are the amplitudes of the transmitted signals with and without the presence of solids, respectively) in the autoclave at a constant temperature of 265 °C and with a constant stirring speed and without nitrogen flow. The A/A_o ratio decreased as the concentration of solids increased from 1 wt.% to 25 wt.%. In general, the amplitude of the transmitted ultrasonic signals decreased as the solids concentration increased. When an ultrasonic pulse is sent across the slurry reactor, the amplitude of the pulse is reduced when it strikes a solid in the slurry because the pulse is partially scattered and absorbed. The scattering and viscous effects are often the predominant forms of attenuation in heterogeneous materials. The dominating mechanism depends on the range of ka where k is the ultrasound wavenumber and a the radius of the particle. The scattering regime dominates when $ka \gg 1$ while the viscous regime governs for small particle size and lower frequencies ($ka < 1$) [21,22]. For the present study, the ka ranges between 0.18 and 0.68. Thus, the viscous effects dominate over scattering. The latter occurs when some of the ultrasonic wave incident upon a discontinuity in a material, for example, a solid particle, is scattered in directions which are different from that of the incident wave. The unscattered pulse is partially transmitted through the solid at the sound velocity of the solid to the receiver. The amplitude of the transmitted portion of the pulse is measured by the receiver located on the opposite side of the autoclave. The more particles present in the path, the

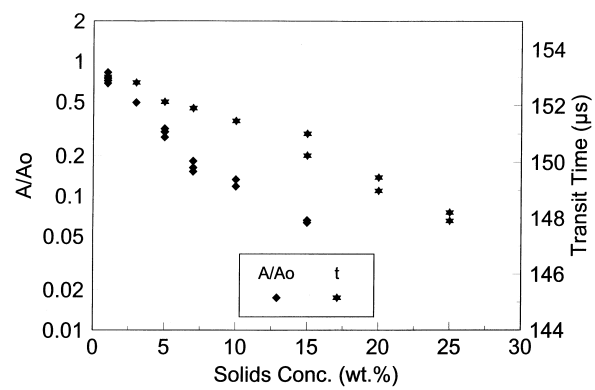

Fig. 4. Amplitude ratio and transit time as a function of solids concentration (wt.%) in FT-200 wax.

less transmitted pulse will be detected. The amplitude ratio of the pulse is inversely proportional to the quantity of solid particles present in the path. Therefore, the measured amplitude of the sound wave transmitted through a slurry mixture is expected to be a strong function of the concentration of solids. Fig. 4 also presents the effect of the solids concentration on the transit time, t_b. The transit time decreased as the solids concentration increased in the reactor. It was approximately 152.125 μs with 5 wt.% of solids, but decreased to approximately 148 μs as the solids concentration increased to 25 wt.%. When an ultrasound wave strikes the boundary between the FT-200 wax and solids, the acoustic impedances of the two media are different and the ultrasound wave will be partially reflected, absorbed, and transmitted. The reflected wave travels back through the wax at the same velocity (844 m s^{-1} for FT-200 wax at 265 °C). The transmitted wave continues to travel through the solids at the sound velocity of the new medium (5448 m s^{-1} for fused silica at 265 °C estimated from Lynnworth [5]). This explains the decrease in transit time as the concentration of solids increased. Furthermore, the concentration of the solids in the slurry greatly affects both amplitude ratio and transit time, t_b, of an ultrasonic signal. The transit time of the transmitted ultrasonic pulse should depend on the quantity of solid particles present in the path. The higher the concentration of solid particles present in the path, the shorter the transit time that would be observed.

Fig. 5 presents the transit time versus the nitrogen flow up to 300 ml min^{-1} at two different solid concentrations, 3 and 25 wt.%. The solids concentrations greatly affect the transit time. It was approximately 152.8 μs at 3 wt.% then decreased further to approximately 148 μs as the solids concentration increased to 25 wt.%. This can be understood by assuming that the presence of solid particles in the path of the sound wave reduces the transit time since the velocity of sound is faster in the solid than in the liquid. The transit time profiles in the range of nitrogen flow studied were rather uniform at both solid concentrations. Thus, one can infer that the variation of nitrogen flow in the reactor did not have significant effects on the observed transit time within the flow conditions studied. The observed approximately constant transit time at any given constant solids concentration with varying nitrogen flows illustrates that the detected portion of the sound is not transmitted through the gas. Separate tests with the stirrer and nitrogen bubbles in the absence of beads confirm that their presence alone causes no effects on the transit time (Fig. 3).

Fig. 6 further illustrates the fractional change in transit time $\Delta t/t_o$ ($\Delta t = t_a - t_b$) as a function of solids concentration in the absence of nitrogen flows in the stirred reactor. The fractional change in transit time (time ratio) increased as the solids concentration increased. A linear regression analysis was conducted on the collected data. The best fit linear line was also plotted in Fig. 6. The reliability of the regression, R^2 value, is 0.978. For the case of cube root dependence on ω, the R^2 value is 0.781. It is clearly indicated that the fractional change in transit time, $\Delta t/t_0$, has a linear relationship with the concentration of solids. In our previous studies [16,17] under nitrogen/water/glass beads system, a simple model has been proposed for the propagation of sound in very dilute slurries. Our model predicts that the change in time ratio $\Delta t/t_0$ is given by the relation

$$\Delta t/t_0 = \alpha\omega^{1/3}(1 - v_l/v_s) \quad (3)$$

Here α is a constant for particles of a given composition and v_l and v_s are the speed of sound in the liquid and solid, respectively. ω is the solid concentration fraction. This expression does not give the correct dependence on solids concentration nor its magnitude.

Urick [23] has proposed a phenomenological approach to correlate the solids concentration and velocity of sound through the medium. The coupled-phase model of suspensions approaches have also been investigated by Harker and Temple [24]. The theoretical multiple scattering approach of ultrasonic propagation of polystyrene spheres in water and glass spheres in epoxy systems has been studied by Anson and Chivers [4]. These approaches can be applied to further theoretical study.

The results from our study are in qualitative agreement with those previously reported by Okamura et al. [15]. Our results with molten wax are similar to the previous results from the nitrogen/water/glass beads system [16,17]; though our previous results give larger values of time ratio at a given

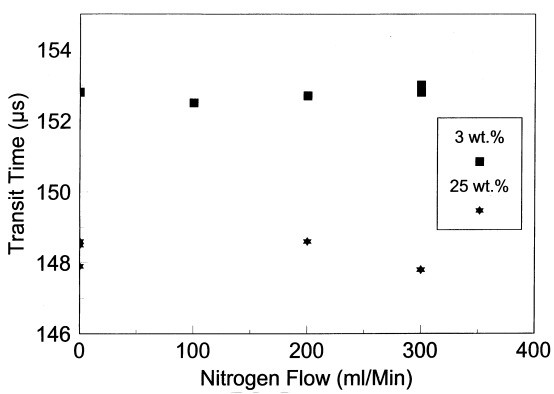

Fig. 5. Transit time as a function of nitrogen flow in FT-200 wax at three different solids concentrations.

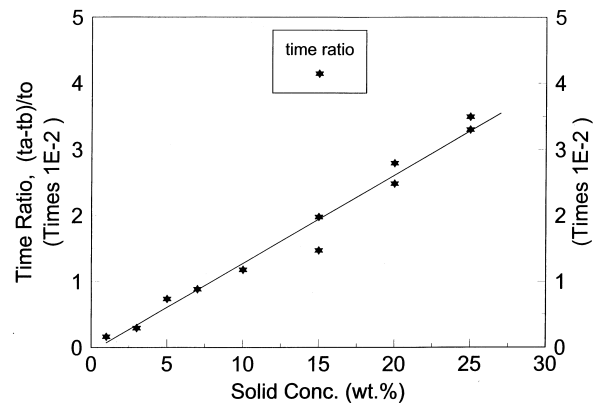

Fig. 6. Time ratio as a function of solids concentration (wt.%) in FT-200 wax.

solids concentration fraction than our current nitrogen/FT-200 wax/glass beads data. A possible explanation of the discrepancy could be that the geometry of our previous apparatus, nitrogen/water/glass bead system at ambient conditions differs considerably from that employed in the current study (autoclave reactor, nitrogen/FT-200 wax/glass beads system, and 265 °C). In general, both the amplitude and the fractional change of transit time (or time ratio) were affected by the variation of solids concentrations. It appears that the time ratio depends mainly on the solids concentration. The variation of nitrogen flow has little effect on the observed fractional change of transit time (or time ratio) within the flow conditions studied. Based on these results, it can be concluded that the ultrasonic technique has potential applications for solids concentration measurements in three-phase slurry reactors at high temperatures.

4. Conclusion

The results presented in this study led to the conclusion that both the amplitude and the transit time of an ultrasonic signal are influenced by the variation of solids concentration in molten FT-200 wax. The variation of nitrogen flow has little influence on the observed transit time within the conditions studied. The ultrasonic technique has potential applications for solids concentration measurements in three-phase slurry reactors at elevated operating temperatures.

5. Nomenclature

a	bubble or particle radius (m)
A	amplitude of the transmitted signal
A_o	amplitude of the transmitted signal in the absence of nitrogen or solid
k	the wavenumber (k) of the ultrasonic wave which surrounds the bubble or particle ($k=2\Pi/\lambda$)
$f(d_b)$	a function dependent on the Sauter mean diameter
t_a	arbitrary first distinct zero crossing time in liquid (s)
t_b	arbitrary first distinct zero crossing time in the presence of solid in slurry (s)
t_o	arbitrary travel time of the sound wave between the transmitter
v_l	speed of sound in liquid (m s^{-1})
v_s	speed of sound in solid (m s^{-1})
Δt	equals (t_a-t_b) (s)
$\Delta t/t_o$	time ratio
x	the travel distance in the path (m)
R^2	the reliability of the regression
$S()$	the scattering coefficient

Greek letters

α	a constant for particles at a given composition
λ	wavelength (m)
ϵ	void fraction
ω	solids concentration fraction
Γ	volumetric interfacial area
ρ_l	fluid density (kg m^{-3})
ρ_s	particle density (kg m^{-3})

6. Disclaimer

Reference in this report to any specific commercial product, process, or service is to facilitate understanding and does not necessarily imply its endorsement or favoring by the United States Department of Energy.

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