

MIXING TIMES IN WATER-BASED POLYSACCHARIDE SYSTEMS IN CYLINDRICAL VESSELS WITH VARIOUS IMPELLERS[†]

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[†]This paper is dedicated to the late Professor Dr. Drago Kolar

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Abstract

Measurements of mixing times for water-based polysaccharide systems were carried out in two flat-bottomed cylindrical plexiglass vessels. Vessels had different dimensions and were equipped with various impellers. Helical ribbon impeller was used for mixing polysaccharide systems with higher viscosity. Mixing times were determined with thermal response technique and compared with flow visualisation method. Rheological examinations were performed by using controlled stress and controlled rate rheometer. Rheological behaviour of polysaccharide systems was investigated under steady and oscillatory shear conditions at different temperatures.

Introduction

Rheologically complex fluids occur in many chemical process industries. Mixing of such fluids is very complicated because the rheological properties depend on shear action. Different rheological complexities that can develop during mixing have a profound effect on a mixing process. For example shear dependent viscosity or viscoelastic behaviour can markedly affect mixing performance. It has already been reported in literature that elasticity alters power requirements and modifies flow patterns. Collias and Prud'homme¹ showed that elasticity substantially increases the torque required for mixing. They also obtained a correlation for extra torque required to mix an elastic fluid. It was also reported that fluid with pseudoplastic behaviour creates small circulation paths around blades and substantially decreases the circulation flow rate.²

Very simple mixing equipment is composed of vessel, impeller and a motor. A wide variety of vessels and impellers differ in diameter, height, shape and other geometrical parameters. Each impeller has its own characteristics and its own area of application. Turbine impellers are usually used for turbulent mixing of fluids with viscosities up to about 10 Pas, depending on the scale, application and process

requirements. On the other hand multiple large diameter axial impeller systems are used for mixing highly viscous materials in laminar flow regime. The application of helical ribbon impellers turned out to be very successful for mixing highly viscous (of about 50 Pas and higher)³ fluids usually with non-Newtonian behaviour.

Mixing performance can be evaluated by measuring power requirements, mixing and circulation times or other similar parameters. Mixing time is in general defined, as time needed to reach a certain degree of homogeneity. The concepts of mixing time and classical techniques to measure it are used to describe macro-mixing processes. The most common methods to determine mixing time are thermal response technique, chemical decolourisation method, conductivity measurements and flow visualisation. In thermal response technique a small amount of a heated sample is poured instantly into the vessel. The complete homogenisation is indicated by the disappearance of temperature difference within the vessel caused by the heated sample tracer. Thermal response technique is very practical and useful because it can be repeated several times. During the measurements the rheological properties of test fluids does not change drastically even when the fluids are thermo-sensitive. This is due to the fact, that the amount of heated sample is so small that the changes of global temperature inside the vessel are less than about 0.5 °C.

The rheological response of complex fluids to an imposed shear stress depends on the magnitude of shearing conditions. Mixing studies of complex fluids must thereby include a consideration of the range of shear rate and/or shear stress during the process. To estimate shear rates within the mixing vessel empirical and theoretical approaches have been proposed in literature. In the approach based upon Couette flow analogy the flow produced by an impeller inside a stirred vessel can be considered similar to that produced by a rotating cylinder (impeller) inside another cylinder (vessel). According to this approach a relationship between the average shear rate, $\dot{\gamma}$, around the impeller, and the impeller rotational speed, N , was postulated by Otto and Metzner:⁴

$$\dot{\gamma}_{av} = K_s N \quad (1)$$

where K_s is a parameter of proportionality and depends on geometry of the impeller.

Chavan and Ulbrecht,^{5,6} studied power consumption for mixing power law fluids with helical ribbon impellers. For laminar region they derived a K_S function by following the extended Couette flow analogy:

$$K_S = 4\pi \frac{\left[(D_T / d_e)^2 - 1 \right]^{1/n-1}}{\left\{ n \left[(D_T / d_e)^{2/n} - 1 \right] \right\}^{n/n-1}}, \quad (2)$$

where D_T is a diameter of the vessel, d_e is an equivalent inside diameter and n is flow behaviour index. The parameter d_e is based solely on the geometry of helical ribbon impeller and is given by:

$$\frac{d_e}{D} = \frac{D_T}{D} - 2 \frac{W}{D} / \ln \left\{ \frac{(D_T / D) - 1 + 2(W / D)}{(D_T / D) - 1} \right\} \quad (3)$$

Reynolds number is usually used to characterise the flow regime and is defined as:

$$\text{Re} = \frac{\rho N D^2}{\eta}, \quad (4)$$

where ρ and η represent the fluid density and viscosity respectively, N is the rotational speed of the impeller and D its diameter. Depending on the impeller speed and viscosity of the fluid mixing regime can be laminar or turbulent. Below $\text{Re} = 10$, the viscous effects predominate and mixing regime is laminar. Turbulence threshold is, depending on the type of impeller, comprised between $\text{Re} = 10^3$ and $\text{Re} = 10^4$. Between laminar and turbulent regime is the intermediate mixing region called transition regime. When mixing low viscosity fluids turbulence is the main physical mechanism responsible for mixing while turbulence can hardly be achieved when viscosity of the fluid is very high.

In the present work mixing of rheologically complex fluids was studied. Measurements of mixing times and flow visualisations were used for describing mixing performance of different mixing systems.

Materials and methods

Equipment

Two geometrically different mixing systems were used. First cylindrical vessel with a diameter of 0.3 m was equipped with four baffles. All the impellers mounted in this vessel had a diameter of 0.1 m (1/3 of the diameter of the vessel). The second vessel of 0.185 m diameter had no baffles. A helical ribbon impeller with a diameter of 0.16 m was used only in this vessel. Both vessels were flat-bottomed plexiglass cylinders. The ratio between height of the liquid and the diameter of the vessel was during all the experiments kept constant and equal to 1.

Fluids and Rheological Characterisation

Aqueous solutions of two different polysaccharides: carboxymethyl cellulose (CMC) and xanthan were used. The solutions were prepared at different concentrations: CMC at 1 wt.% and 1.5 wt.% and xanthan at concentration 2 wt.%. All aqueous polysaccharide systems were prepared by dissolving powdered polysaccharide in distilled water at room temperature.

The rheological tests were performed using the controlled rate rheometer HAAKE CV 20 and the controlled stress rheometer HAAKE RS 150 equipped with different sensor systems. Steady and oscillatory shear tests were performed at different temperatures since the mixing time was measured with thermal response technique. To determine the influence of mixing on the flow properties the same rheological tests were done before, during and after mixing.

The flow behaviour in the shear rate range existing inside the vessel during mixing process was described by the power law model:⁷

$$\tau = K\dot{\gamma}^n, \quad (5)$$

where K and n are consistency and flow behaviour index, respectively. The decrease of apparent viscosity η with increasing shear rate, $\dot{\gamma}_{av}$ is therefore expressed as:

$$\eta = \frac{\tau}{\dot{\gamma}} = K\dot{\gamma}^{n-1} \quad (6)$$

For used polysaccharide systems the values of parameters K and n are for listed in Table 1.

Table 1: Rheological properties of used polysaccharide systems (at 25 °C)

Polysaccharide system	K [Pas ⁿ]	n [/]
CMC (1 wt.%)	2.33	0.57
CMC (1.5 wt.%)	6.54	0.52
xanthan (2 wt.%)	8.24	0.27

Measurement of mixing time

The mixing times were determined by thermal response technique. After the injection of small amount of heated fluid the change of temperature was followed by using two first order thermistors with very low time constant. The sample was heated to 50 °C and always injected in the vessel at the same injection point. The initial temperature in the mixing vessel was in the range from 20 to 25 °C. During all experiments the positions of two thermistor probes were kept constant (Figure 1). Mixing times were measured as a function of impeller speed, impeller type and rheological properties of the fluid.

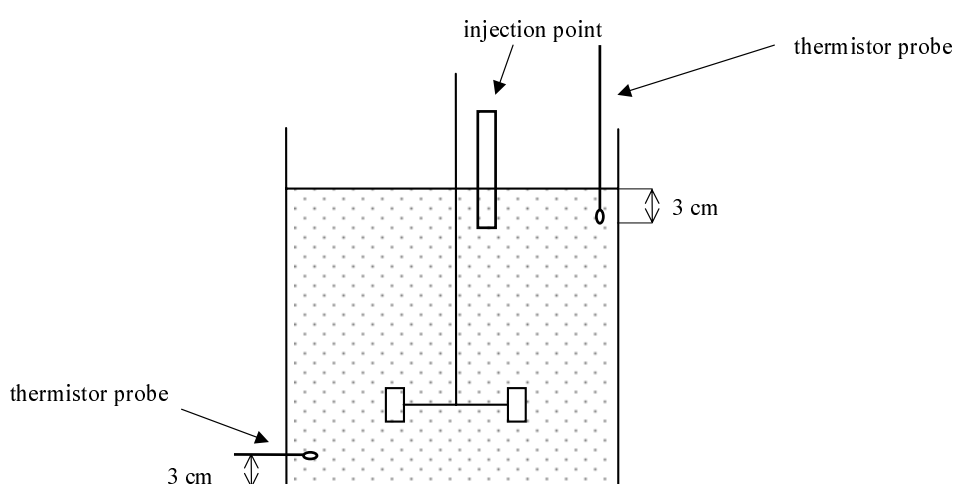


Figure 1: Position of the thermistor probes and localisation of the injection point

From the response curve two different times were determined: a mixing time, t_{mix} , as the time when the response of thermal probe stabilised at 5% of the maximum value and a dead time, t_c , as the time between the injection of the hot fluid and its first detection at the thermistor probe (Figure 2).

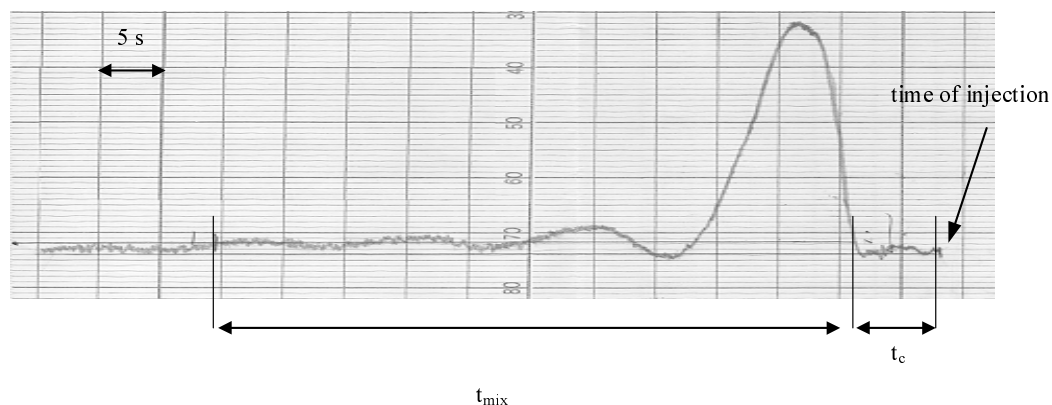


Figure 2: Example of a thermal probe response obtained during mixing time experiments

The average shear rate, $\dot{\gamma}_{av}$, around the impeller, was determined with Otto-Metzner equation (1), where K_s values for helical ribbon impeller were obtained from the literature with the expression (2). K_s depended on the power law index n , but the dependence wasn't strong (Figure 3).

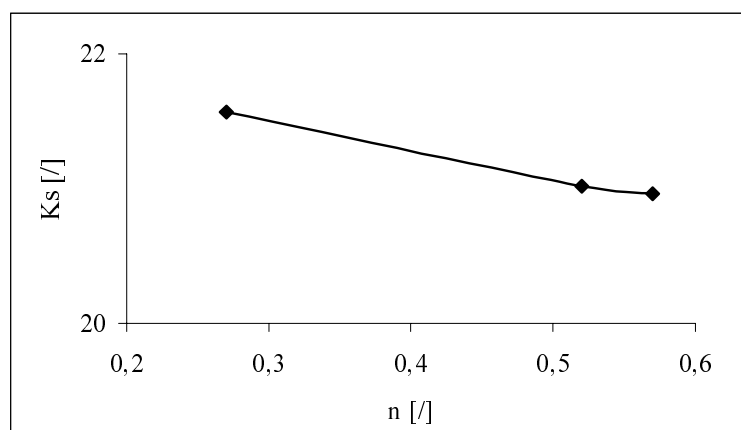


Figure 3: K_s values calculated from the equation (2) as a function of n

To compare actual homogenisation time with mixing time, determined with thermal response technique, visualisation method was used. Instead of heated sample a small amount of coloured sample (KMnO_4) was injected at the same spot as the heated sample in the thermal response technique. The flow induced by the impeller was followed by taking photographs at different times.

Results and discussion

Degradability of examined polysaccharides that may occur during mixing process, was checked by the rheological measurements. The same rheological tests were performed before, during and after mixing process. The rheological results showed that the mixing process had no influence on the stability of the mixing media. The flow curves of investigated samples at 25 °C are shown in Figure 4. The polysaccharide systems differed in rheological properties. Under destructive shear conditions all the samples exhibited pseudoplastic behaviour. The shear thinning behaviour was more noticeable for xanthan system (Figure 4 and Table 1). Under non-destructive shear conditions CMC systems exhibited the rheological properties characteristic for polymer solutions whereas xanthan system exhibited more pronounced viscoelastic behaviour, which is usually found in weakly structured materials.

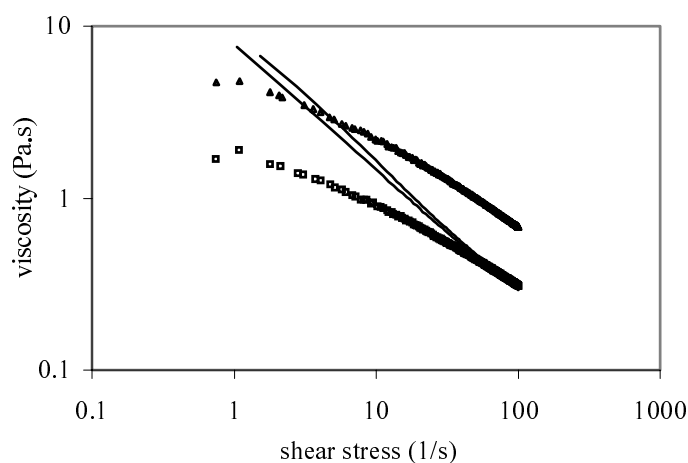


Figure 4: Rheological characterization of examined fluids at 25 °C (\square - 1 wt.% CMC, Δ - 1.5 wt.% CMC, solid line - 2 wt.% xanthan)

Temperature dependence of examined polysaccharide systems was studied to investigate possible changes that could occur due to heating of the sample during thermal response technique. Tests showed that the rheological behaviour at higher temperature remained pseudoplastic and time independent. Furthermore the amounts of heated samples were very small compared to entire fluid in the vessel therefore the temperature of the heated sample did not considerably affect the properties of the fluid in the vessel.

Mixing times were measured and compared among different impeller systems in both vessels. With the existing experimental set-up the mixing times for the system with low apparent viscosity (1 wt.% CMC) were possible to determine only in the range of $Re = 100$ to $Re = 1000$. In this Re range mixing time strongly depended on the impeller type. Rushton turbine generates radial flow pattern, while propeller generates typical axial flow pattern. Mixing times obtained with the Rushton turbine were much shorter than those obtained with the propeller. The difference between mixing times was more noticeable at lower Re -numbers.

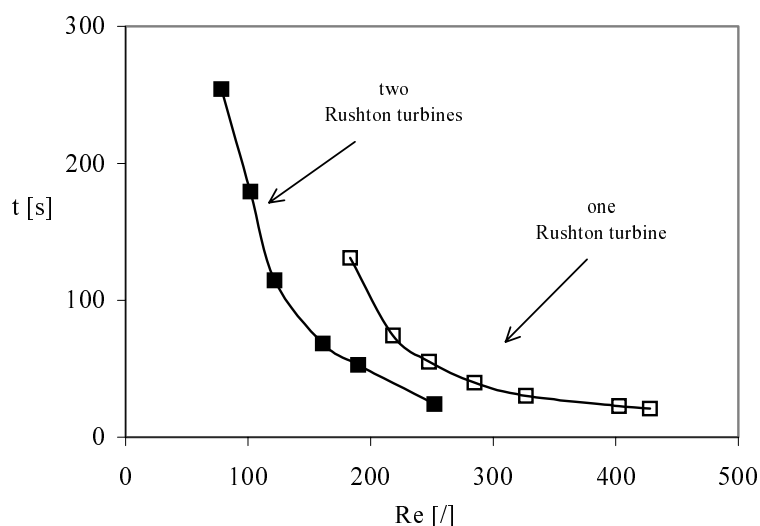


Figure 5: Effect of the number of impellers on mixing with 1 wt.% CMC in 0.3m diameter vessel

To facilitate better homogenisation two impellers were mounted on one shaft. As illustrated in Figure 5 the mixing times depended on the number of impellers. With two Rushton turbines mixing times were determined at lower values of Reynolds number. In

the case of mixing with one Rushton turbine a rise of stagnant zones was visually observed. In these zones mixing was poor and determination of mixing time was not exact. Application of two Rushton turbines decreased the stagnant zones and improved homogenisation. When more viscous systems (1.5 wt.% CMC and 2 wt.% xanthan) were mixed with single impellers, stagnant zones were observed more frequently. Hence, for these systems, determination of mixing times was not possible.

The combination of two impellers that cause different flow patterns (radial, axial) gave better results than impeller system of two identical impellers. Figure 6 shows the dependence of dimensionless mixing time on the type of two impellers mounted on the same shaft. Mixing times were compared between two Rushton turbines and one Rushton turbine with one propeller on the same shaft. For both impeller systems the mixing times were determined almost in the same range of Reynolds number. Over the whole studied range mixing with two different impellers gave shorter mixing times with more pronounced difference at lower Reynolds numbers.

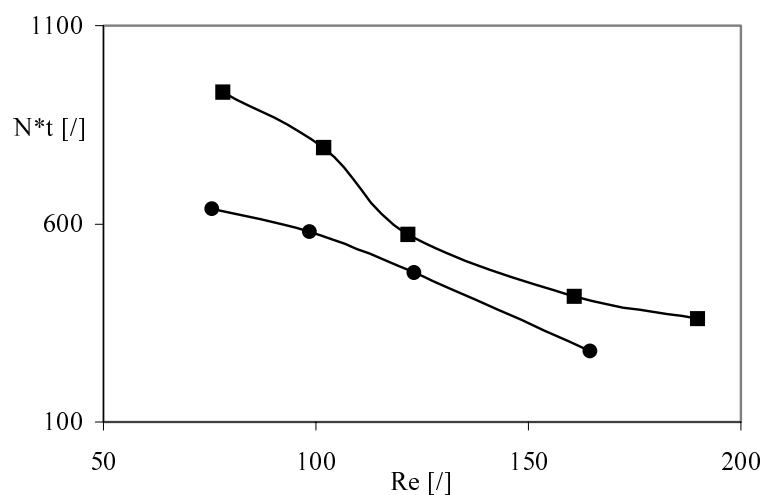


Figure 6: Mixing with two impellers on the same shaft (1 wt.% CMC in vessel with 0.3 m diameter): ■ two Rushton turbines, ● Rushton turbine + propeller

Figure 7 presents flow visualisation method for 1.5 wt.% CMC, using two Rushton turbines in 0.19 m diameter vessel. Small amount of KMnO_4 was added as a tracer fluid at the liquid surface and slowly mixed in. The impeller was rotating at $N =$

7.5 s^{-1} . The photographs were taken before the tracer addition and after 1, 15 and 305 s. The upper turbine pulled down the tracer 1 s after the addition (Figure 7 b). The tracer was mixed only with the upper turbine and, hence, two separated regions were formed (Figure 7 c). Later also the bottom turbine pulled the tracer down through the centre and the tracer was mixed with both turbines. The border between two separated regions disappeared after 275 s. At the time that was determined as mixing time with thermal response technique (Figure 7 d) this two regions were homogenised, but a thin stagnant layer at the bottom of the vessel was observed. The mixing process was followed also at longer times (longer than determined t_{mix}) and the bottom layer was still present. In this layer the mixing proceeded extremely slowly.

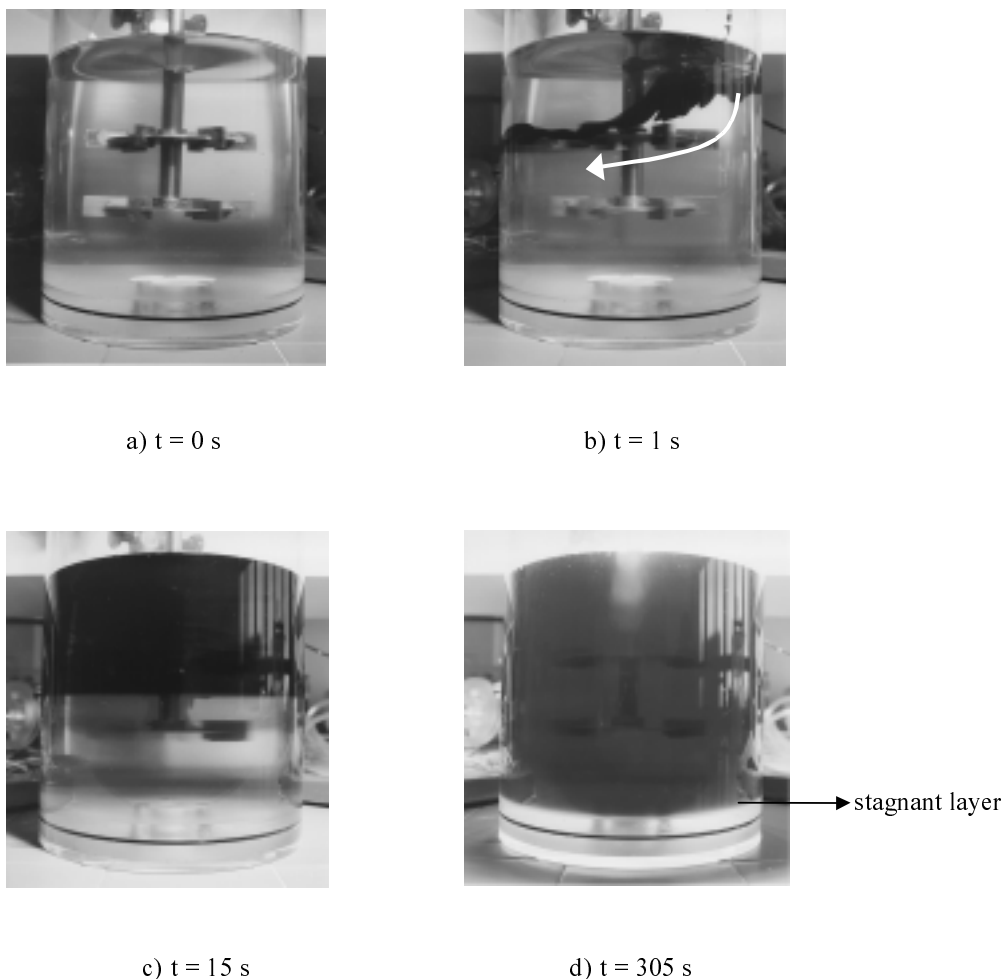


Figure 7: Results of mixing time experiment with two Rushton turbines in the 0.19 m diameter vessel

More viscous fluids (1.5 wt.% CMC and 2 wt.% xanthan) were mixed also with helical ribbon impeller (Figure 8 a) which should be used as an alternative to multiple impeller systems.⁸ Helical ribbon impeller had a diameter very close to the inside diameter of the vessel and hence guaranteed liquid motion throughout the vessel. Mixing of 1.5 wt.% CMC with helical ribbon impeller is presented in Figure 8.

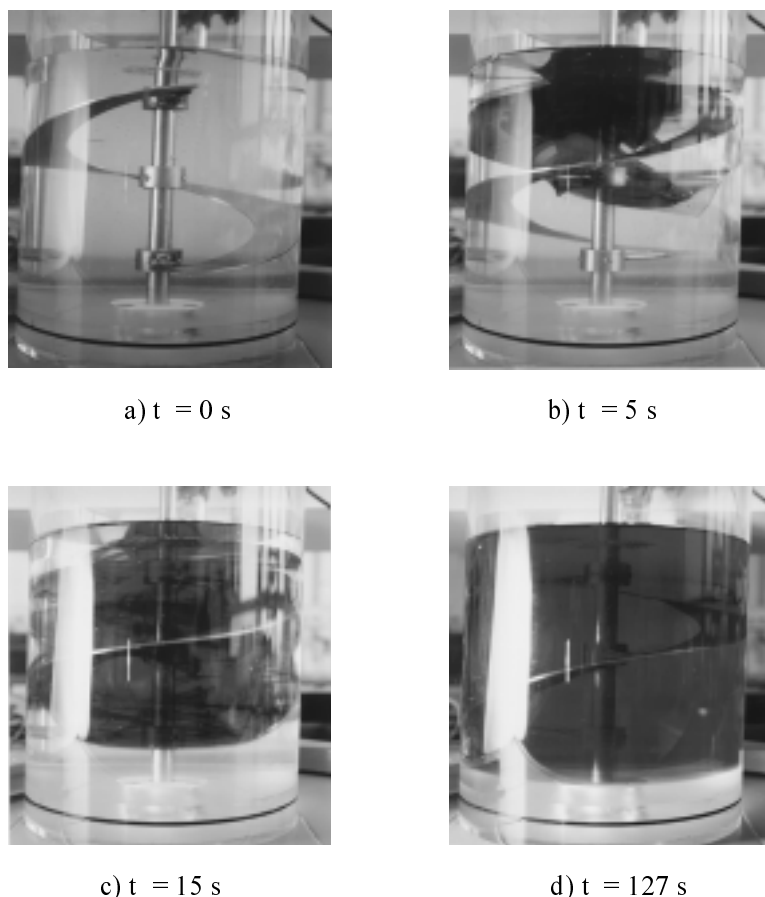


Figure 8: Results of mixing time experiment with helical ribbon impeller in the 0.19 m diameter vessel

The photographs in Figure 8 were taken at the beginning of mixing ($t = 0$), at 5, 15 and 127 s after the addition of the coloured sample. Helical ribbon impeller was rotating at 1 s^{-1} . The flow pattern created by helical ribbon impeller can be seen from the photographs. Also for this impeller (similar as for two Rushton turbines, Figure 7) the mixing process was followed longer than mixing time $t_{\text{mix}} = 127\text{s}$ which was determined with thermal response technique. A thin stagnant layer at the bottom of the vessel was

observed also in this case. The thickness of the layer decreased very slowly. After 330s the layer was 3 mm thick, and after 870 s it was still 1 mm.

Figure 9 presents the difference between mixing of two viscous liquids with helical ribbon impeller. In both cases mixing was performed at lower Reynolds numbers as when the other impellers were used. The mixing times for this impeller system strongly depended on the rheological properties of the fluid. Shorter mixing times were obtained for the system with less pseudoplastic behaviour (1.5 wt.% CMC).

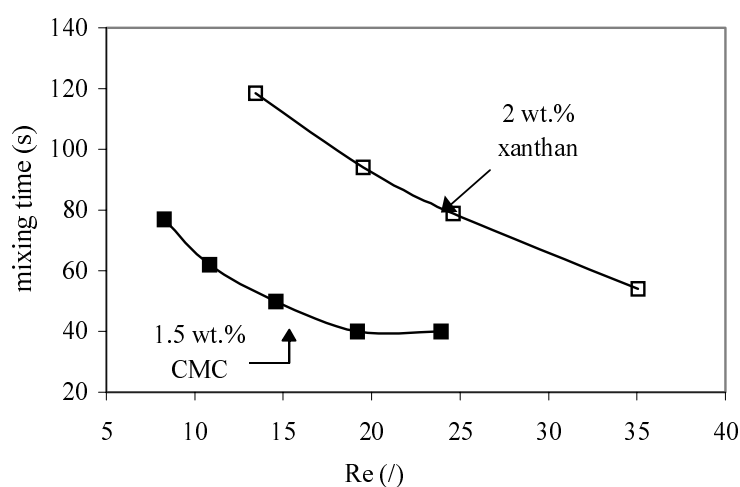


Figure 9: Mixing time with helical ribbon impeller in vessel with 0.19 m diameter

Conclusions

The mixing performance of two different mixing systems was studied by measuring mixing time. The experimental results were obtained for two mixing systems equipped with different impellers. It was shown that the mixing time and, consequently, the mixing performance depended on the type and number of impellers, rheological properties of the fluid and the size of the vessel. With single impellers good homogenisation was not achieved throughout the vessel. Stagnant zones occurred more often in more viscous fluids, where mixing with single impeller was not sufficient.

The use of double impeller systems improved mixing efficiency, but some stagnant zones still occurred, especially in the case of highly viscous fluids. It turned out that for

highly viscous fluids the use of helical ribbon impellers gave better results and enabled effective mixing in laminar flow regime.^{3,8}

We confirmed that the thermal response technique is very useful for evaluating mixing performance. The repeatability of the method is very good (the estimated error is within the range of $\pm 5\%$), but the results can sometimes lead to wrong conclusions about flow patterns, especially with highly viscous fluids. Visualisation method on the other hand enables observation and determination of the real conditions during mixing. The existence of stagnant zones can be identified. The combination of both methods enable the design of another, better mixing system that could decrease stagnant zones and improve mixing efficiency.

Nomenclature

D	diameter of the impeller	(m)
D_T	diameter of the vessel	(m)
d_e	equivalent inside diameter	(m)
K	consistency index	(Pa ⁿ)
K_s	defined by Equation (2)	(/)
N	impeller rotational speed	(s ⁻¹)
n	flow behaviour index	(/)
Re	Reynolds number	(/)
W	width of the impeller blade	(m)
$\dot{\gamma}_{av}$	average shear rate	(s ⁻¹)
η	fluid viscosity	(Pas)
ρ	fluid density	(kg/m ³)
τ	shear stress	(Pa)

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Povzetek

Čase pomešanja vodnih polisaharidnih sistemov smo proučevali v dveh cilindričnih posodah z ravnim dnom. Posodi sta imeli različne geometrijske dimenzije, opremljeni pa sta bili z različnimi mešali. Za mešanje visoko-viskoznihs polisaharidnih sistemov smo uporabili tračno mešalo. Čase pomešanja smo določevali s termično metodo, tokovne značilnosti pa smo vizualno opazovali in fotografirali. S pomočjo reoloških meritev, ki smo jih izvedli s pomočjo rotacijskega reometra z nastavljivo strižno hitrostjo in rotacijskega reometra z nastavljivo strižno napetostjo pred, med in po mešanju, smo študirali degradibilnost polisaharidov. Reološko obnašanje polisaharidnih sistemov pri destruktivnih in nedestruktivnih strižnih pogojih smo proučevali pri različnih temperaturah.