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The effect of operating conditions on a reaction calorimeter[1]

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Abstract

We have determined the dependence of the measured overall heat transfer coefficient, U, on the agitation speed, the volume of fluid inside the reactor vessel and the calibration time by designing 36 experiments. The matrix of these experiments was prepared using software for the design of the experiments.

The overall heat transfer coefficient, U, for the above 36 experiments was calculated based on both the heat transfer area at zero agitation and the observed area after the liquid level climbed up the wall due to the fluid being stirred. The analysis of the results shows the dependence of U, based on either initial area or observed area, on the agitation speed and fluid volume. Calibration time was found not to influence the value of U.

Keywords: Agitation; Calibration; Coefficient; Heat; Mettler

Nomenclature

A	area of contact of liquid and the reactor wall at zero agitation/m^2
A_{act}	area of contact of liquid and the reactor wall/m^2
b	a fluid property factor defined in the Wilson method/W m^{-2}°C^{-1}
$C1$	a dimensionless geometry factor defined in the Wilson method
c_p	heat capacity of the liquid in the reactor /J kg^{-1}°C^{-1}
D	inside diameter of the reactor/m
d	impeller diameter/m

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f	a factor defined in the Sieder Tate Correlation
g	gravitational acceleration/m s^{-2}
h_c	a combined scale resistance defined by Eq. (2)/(W m^{-2}°C^{-1})$^{-1}$
h_i	reactor-side film heat transfer coefficient/W m^{-2}°C^{-1}
h_j	jacket-side film heat transfer coefficient/W m^{-2}°C^{-1}
h_{si}	scale resistance on reactor side/(W m^{-2}°C^{-}1)$^{-1}$
h_{sj}	scale resistance on jacket side/(W m^{-2}°C^{-1})$^{-1}$
k	heat conductivity of the bulk liquid in the reactor/W m^{-1}°C^{-1}
k_w	reactor wall heat conductivity/W m^{-1}°C^{-1}
n	agitation speed/rpm
n_0	reference agitation speed/rpm
U	overall heat transfer coefficient based on area A/W m^{-2}°C^{-1}
U_{act}	overall heat transfer coefficient based on area A_{act}/W m^{-2}°C^{-1}
X_w	reactor wall thickness/m

Greek letters

ρ	density of the liquid in the reactor/kg m^{-3}
μ	viscosity of the bulk liquid in the reactor/N s m^{-2}
μ_w	viscosity of the liquid at the reactor wall/N s m^{-2}

Dimensionless numbers

Re	Reynolds Number $= d^2 n\rho/\mu$
Pr	Prandtl Number $= c_p\mu/k$

1. Introduction

A calorimeter is small-scale automated jacketed reactor equipped with a precise temperature controller and is capable of heat balancing. The liquid inside the reactor may be heated or cooled by the jacket fluid while being stirred using different types of agitators, driven by a motor with adjustable speed control. In an automated calorimeter, reactor (T_r) and jacket (T_j) temperatures are continuously measured and a powerful thermostat allows rapid adjustment of jacket temperature to maintain a desired reactor temperature profile.

In order to calibrate the calorimeter, an electrical heater is provided inside the reactor that can introduce a known thermal load to the fluid. This heat input leads to a difference between the reactor and the jacket temperatures, ΔT, that is continuously measured. The product UA can then be calculated as

$$UA = \frac{\int_0^t Q_c(t)\,\mathrm{d}t}{\int_0^t \Delta T(t)\,\mathrm{d}t}$$

Where A is the heat transfer area and Q_c is determined from the thermal power input.

The product UA may then be divided by the area available to heat transfer, A, to obtain the overall heat transfer coefficient.

Calibration of the calorimeter may be carried out by heating a known volume of liquid inside the calorimeter at a desired agitation speed. Heat is transferred through the area where the liquid contacts the reactor wall; this may vary with agitation speed because the liquid level tends to climb up the wall. The volume of the liquid in the reactor and the calibration heat input rate may affect the outcome of the calibration. This article describes a study on the effect of calibration time, agitation speed and volume of the liquid in the reactor on the measured overall heat transfer coefficient.

2. Experimental runs

A matrix of operating conditions was prepared for a number of experimental runs with the Mettler RC1. This was done using software for experimental design. The matrix included 36 experiments (24 replicates) using three variables: calibration time (15,22.5 and 30 min), volume of fluid in the reactor (700, 1200 and 1700 ml) and agitation speed (75, 163 and 250 rpm).

The experiments were carried out using a Mettler RC1 calorimeter with a glass reactor type AP01 and a standard anchor stirrer. Heat was added to toluene in the reactor by the electric heater and removed by the jacket fluid. A constant reactor temperature of 30°C and a constant difference between the reactor and the jacket temperatures, ΔT, were established. The value of UA for each run was determined by the Mettler RC1 from the power input and ΔT. The overall heat transfer coefficient, U, was then determined based on A the area corresponding to the volume of fluid in the reactor vessel.

During each run, due to agitation, the liquid level may rise up the wall causing the area available for heat transfer to increase. The level of the liquid was observed through the reactor glass wall in each run and, based on that, an actual area available to heat transfer, A_{act}, was estimated. The estimate was reasonable since the liquid level did not seem to be wobbly. An actual overall heat transfer coefficient, U_{act}, was determined based on the estimated actual area.

The dependence of U and U_{act} on the three variables, time, volume and speed, was studied. The overall heat transfer coefficient, either U or U_{act}, was found to be dependent on fluid volume in the reactor and agitation speed, but independent of calibration time over the range examined as shown in Table 1.

3. Wilson plot for calorimetry runs

In order to determine the reactor-side and jacket-side heat transfer coefficients, the Wilson method of analysis [1,2] was used to decouple to experimentally measured overall heat transfer coefficient.

Table 1
Effect of calibration time, liquid volume in the reactor and agitation speed on the heat transfer coefficient

Time/min	Volume/ml	Speed/rpm	U/W m^{-2}°C^{-1}	U_{act}/W m^{-2}°C^{-1}
15	700	75	143	135
15	700	75	143	135
15	700	75	143	135
30	700	75	142	134
30	700	75	142	134
30	700	75	141	133
22.5	1200	75	132	129
22.5	1200	75	130	127
22.5	1200	75	132	128
15	1700	75	132	130
15	1700	75	132	130
15	1700	75	132	130
30	1700	75	133	130
30	1700	75	132	130
30	1700	75	133	131
22.5	700	163	187	160
22.5	700	163	188	160
22.5	700	163	187	160
15	1200	163	157	140
15	1200	163	157	140
15	1200	163	157	140
15	700	250	234	157
15	700	250	236	159
15	700	250	229	154
30	700	250	236	158
30	700	250	239	160
30	700	250	234	157
30	1200	250	190	145
30	1200	250	190	145
30	1200	250	191	146
15	1700	250	162	144
15	1700	250	161	144
15	1700	250	161	144
30	1700	250	162	144
30	1700	250	161	143
30	1700	250	161	144

According to the Wilson method, the overall heat transfer coefficient is related to agitaion speed as

$$1/U = 1/h_c + [1/(f\,C1\,b)]\,(n/n_0)^{-2/3} \qquad (1)$$

where $C1 = (d^4 n_0^2/gD^3)^{1/3}$ is a vessel-dependent factor,
$b = (c_p \rho^2 k^2 g/\mu)^{1/3}$ is a fluid properties factor, f is a factor in the form of an empirical equation known as the Sieder Tate Correlation [3] for the inside heat transfer

coefficient given as $h_i = (k/D) f \mathrm{Re}^{2/3} \mathrm{Pr}^{1/3} (\mu/\mu_w)^{0.14}$, and h_c is given by

$$1/h_c = 1/h_{si} + x_w/k_w + 1/h_{sj} + 1/h_j \tag{2}$$

A plot of $1/U$ versus $(n/n_0)^{-2/3}$ is known as a Wilson Plot. A straight line fit of a Wilson plot would give $1/h_c$ as the intercept and $1/f\,C1\,b$ as the slope.

4. Analysis of the results

The reciprocal of the overall heat transfer coefficient, $1/U_{\mathrm{act}}$, was plotted against the dimensionless term $(n/n_0)^{-2/3}$ where n is the agitation speed in rpm and n_0 is an arbitrary reference speed chosen as 60 rpm. The plot is shown in Fig. 1 which indicates a high degree of scatter ($R^2 = 0.695$) among the data. The slope and intercept of the line are 0.002 and 0.0059, respectively. The reciprocal of the intercept is h_c, equal to 171 $\mathrm{W\,m^{-2}\,°C^{-1}}$.

The scale resistances in Eq. (2) may be assumed as $h_{si} = 5700\ \mathrm{W\,m^{-2}\,°C^{-1}}$ and $h_{sj} = 2800\ \mathrm{W\,m^{-2}\,°C^{-1}}$ for the AP01 glass reactor [4]. The reactor wall is glass of 0.005 m thickness and thermal conductivity of 1.2 $\mathrm{W\,m^{-1}\,°C^{-1}}$. Substituting the above numbers in Eq. (2) and rearranging will give an equation for the jacket-side heat transfer coefficient as

$$h_j = h_c/(1 - 0.0047 h_c) \tag{3}$$

Using $h_c = 171\ \mathrm{W\,m^{-2}\,°C^{-1}}$ in Eq. (3) will give $h_j = 871\ \mathrm{W\,m^{-2}\,°C^{-1}}$.

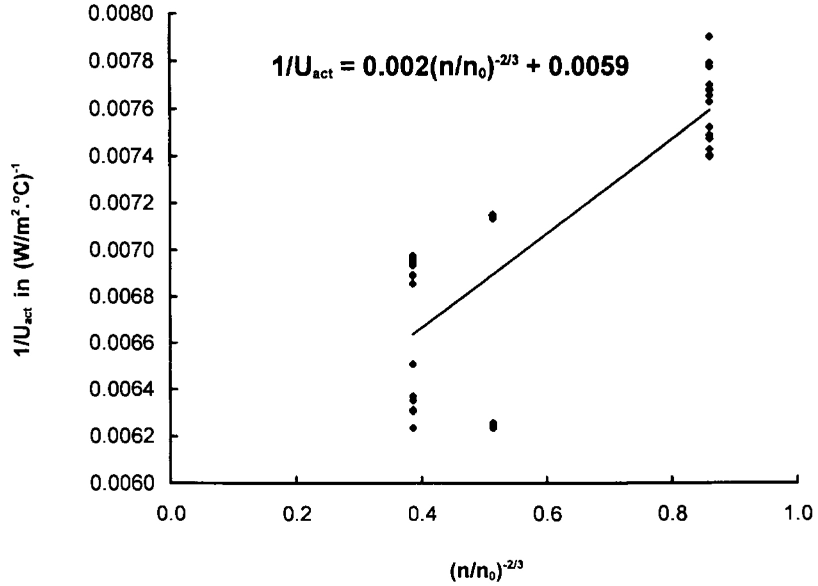

Fig. 1. Wilson plot for all volumes of toluene in the reactor.

The f factor in Eq. (1) is related to slope as

$$\text{Slope} = 1/f\,C1\,b \tag{4}$$

For the reactor AP01, the vessel and impeller diameters are 0.115 m and 0.105 m, respectively. The value of $C1$ was calculated as 0.2. Also the parameter b was calculated for toluene at 30°C as 7500 $\mathrm{W\,m^{-2}\,{}^{\circ}C^{-1}}$. Therefore, the f factor could be determined from the slope according to

$$f = 1/(1500 \times \text{slope}) \tag{5}$$

Using a slope of 0.002 from Fig. 1 in Eq. (5) will result in an f factor equal to 0.33.

Eqs. (3) and (5) indicate that values of f and h_j are very sensitive to the orientation of the line shown in Fig. 1. A slight change in slope can influence the estimated f since it is multiplied by the very large value of 1500. A similar argument applies to estimating h_j using Eq. (3). Because of the high sensitivity mentioned above, analysis of the scatter of data in Fig. 1 becomes very important.

As previously discussed, the overall heat transfer coefficient, U_{act}, was found to be dependent on the volume of the liquid in the vessel as well as the agitation speed. The Wilson method was applied to each set of data at a constant volume of toluene in the reactor. The result is shown in Fig. 2 where the quality of line fitting is much higher ($R^2 = 0.912$–0.997) than that of the line shown in Fig. 1. However, the quality of fit is better for 1.2 and 1.7 litre volumes than for 0.7 litre as shown in Fig. 2. This may be attributed to the difficulty in estimating A_{act} at lower volumes. The effect of volume becomes much less pronounced for higher liquid volumes as shown in Fig. 2. The

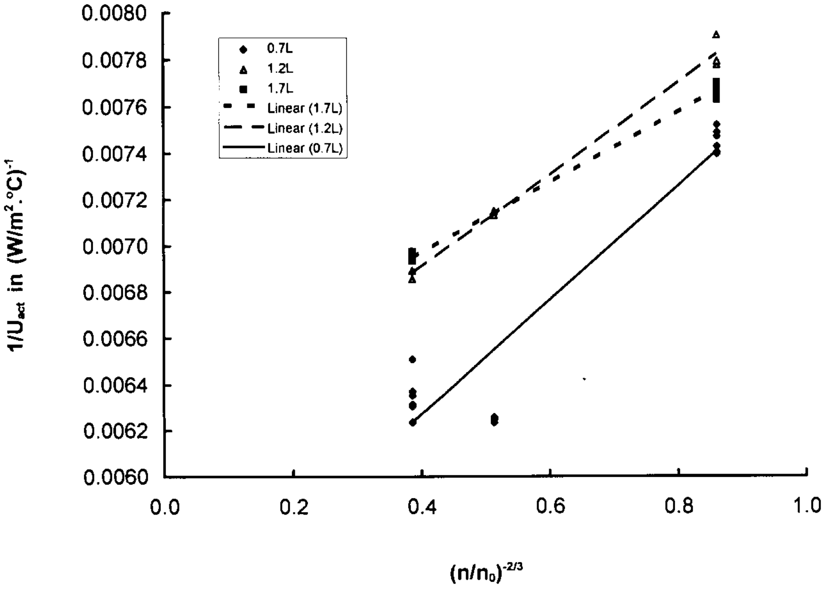

Fig. 2. Wilson plot for different volumes of toluene in the reactor.

Table 2
Values of f factor and jacket-side heat transfer coefficient for the Mettler RC1 reactor AP01

Parameter	Volume of toluene in the vessel at 30°C			Values reported by Choudhury and Riesen [4]
	0.7 l	1.2 l	1.7 l	1.7 l, $T = 35$°C
f	0.27	0.34	0.45	0.48
h_j/W m^{-2} °C^{-1}	1710	704	599	550

values of the f factor and h_j were calculated for three different volumes from the slopes and intercepts of lines in Fig. 2 and compared to the literature data as shown in Table 2.

5. Conclusions

The overall heat transfer coefficient measured in the calorimeter seems to vary with the agitation speed and fluid volume in the reactor. Calibration time was found not to influence the overall heat transfer coefficient. In order to calculate the reactor and jacket-side heat transfer coefficients, the Wilson method of analysis may be used for data points taken at a constant volume of fluid in the reactor.

The values of the heat transfer coefficients determined by the Wilson plot become less dependent on the volume of the liquid in the reactor at higher volumes. Choudhury and Riesen [4] suggested that the AP01 reactor fill should always be more than 1.6 l to ensure a correct observed volume. This is in agreement with the above findings.

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