



3 Investigating the effects of initial oil volume and equilibrium time on the swelling factor using response surface method

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ABSTRACT

The purpose of this study is to investigate the effects of initial oil volume and equilibrium time on the swelling factor during swelling tests. These parameters have not been addressed thoroughly in previous studies. Most investigators used only their own initial oil volume and equilibrium time during their experiments. The present study is focused on investigating the effects of these parameters under various initial oil volumes and equilibrium times according to the design of experiments. The optimum initial oil volume and equilibrium time to yield maximum swelling factor are determined. The initial oil volume and equilibrium time are found to contribute a positive impact to the swelling factor. Based on the results, the maximum initial oil volume is 3.4 cc or 85% of the total cell volume while the optimum equilibrium time is 101 min. The increment of the initial oil volume of .5 cc or 12.5% to 3.4 cc or 85% increases the swelling factor to 6.7% while the increment of the equilibrium time from 30 to 101 min increases the swelling factor to 4.9%. The study also found that the response surface method is appropriate to produce the optimum parameters in order to get maximum swelling factor.

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Introduction

A swelling test is a single contact phase behavior experiment that attempts to measure the CO₂ ability to vaporize or extract an amount of hydrocarbon from crude oils. The importance of this experiment is to know how much the crude oil can swell when in contact with CO₂ at a specific reservoir condition. The test is normally conducted at a constant volume view cell filled with crude oil using high pressure.

Previous researchers used various view cells during the experiment such as 3–14 cc by Tsau, Bui, and Willhite (2010), 140 cc by Hand and Pinczewski (1990), 170 cc by Harmon and Grigg (1988), 111 cc by Wang (1986), and 190 cc by Orr and Silva (1981). Thus, most researchers conducted experiments under their own cell volume. According to the cell volume, a researcher filled the cell with various amounts of oil sample such as 9.0% of total volume done by Wang (1986), 15–53.8% by Tsau et al. (2010), 30% by Harmon and Grigg (1988), and 30–37% by Hand and Pinczewski (1990). The initial oil volume is an important parameter for the experiment and it affects the resulted swelling factor. The smaller the initial oil volume with larger gas phase volume, the greater the amount

of the hydrocarbon that can be extracted from the oil as shown by Tsau et al. (2010). At the lower initial oil volume, the curve displays both oil swelling and shrinkage, while at higher initial oil volume, the curve shows only oil swelling. At the higher initial oil volume, the gas volume in the cell saturates only to the oil as explained by Hand and Pinczewski (1990). However, those researchers did not explain in detail the effects of the initial oil volume and equilibrium time on the resulted swelling factor. Especially, they did not explain the interaction between the parameters and the effects of the interaction on the swelling factor. Results from Hand and Pinczewski's experiment showed swelling and shrinkage phenomena when the initial oil volume is 30%. But, above 37%, the result represented only swelling phenomenon. Tsau et al. (2010) showed different results in their experiment. The initial oil volumes they used were 12, 35, and 54% and the curve shows both swelling and shrinkage phenomena.

When CO₂ is injected into the cell, it does not mix directly but it needs a certain time to reach the equilibrium condition. After the CO₂ and the oil reach the equilibrium condition, the oil reaches the maximum swell. That state is the best condition to measure the oil swelling.

A system is considered in equilibrium when its pressure and vapor density remain constant for several minutes. After each addition of CO₂, the fluid in the cell is circulated for about 20 min and the density is measured such as that done by Harmon and Grigg (1988). After the CO₂ is injected into the cell and it mixes with the oil, the equilibrium time between the two fluids would be between 30 and 60 min after vigorous mixing as suggested by Tsau et al. (2010).

In the present study, the swelling test is conducted under the design of experiments (DOE) using the response surface method (RSM). The purpose of the experimental design is to understand how to conduct and plan the experiment in order to obtain the maximum amount of information from the collected data in the presence of noise and fewer numbers of experiments. The RSM has been widely used in various researches such as constructing the minimum miscibility pressure correlation by Ghomian and Pope (2008), conducting uncertainty analysis of reservoir simulation forecast by Li and Friedmann (2005), history matching by Landa and Guyaguler (2007), optimizing alkaline-surfactant-polymer (ASP) process by Zerpa, Queipo, Pintos, Tillerio, and Alter (2007), and designing operational conditions in heavy oil recovery by Nguyen, Bae, Gunadi, and Park (2014).

The RSM is basically a collection of statistical and mathematical techniques useful to develop, improve, and optimize processes as described by Myers, Montgomery, and Cook (2009). The method has been commonly applied in the petroleum industry since 1990's. Many investigators have used the RSM for solving various petroleum engineering problems as mentioned in the preceding section. The RSM has several design models such as D-Optimal, Full Factorial, Plackett Burman, Central Composite, Box-Behnken, and some others. Some models focus on good prediction of model parameters. Some others focus on good prediction in the design region. D-Optimal focuses on good model parameter estimation based on the notion of the experimental design explained by Myers et al. (2009). D-Optimal model iteratively exchanges design points for candidate points in order to reduce the variance of the coefficient that will be estimated using the design. The regression model is quadratic meaning the design matrix is returned by the optimization algorithm

Table 1. Variables and experimental design levels in the swelling test.

Parameters	Symbol	Coded levels		
		-1	0	+1
Pressure, psi	Pre	600	1500	2500
Temperature, F	Temp	140	145	150
Initial oil volume, cc	Oil	.5	1.5	3.4
Equilibrium time, min	Equ	30	75	120

including linear interaction between parameters and squared terms.

The purpose of this study is to investigate the effects of initial oil volume and equilibrium time on the swelling factor under various pressures and temperatures. This study is unique and different from previous studies because it combines the methods of experiment and response surface methodology in order to investigate effects of the two parameters of interest. The present method shows the interaction between the parameters and the effects of the parameters on the swelling factor. These efforts have not been found to be addressed thoroughly in the literature. The results show some optimum condition for the initial oil volume and equilibrium time to yield the maximum swelling factor.

Methodology

The D-Optimal design is conducted to yield the maximum response of swelling factor from 4 (four) parameters including pressure, temperatures, initial oil volume, and equilibrium time. For each of the parameters studied, high (coded value: +1), middle (coded value: 0), and low (coded value: -1) values are assigned. Set points are selected based on the results shown in Table 1 including variables and experimental design levels in the swelling test. The D-Optimal experimental design shows that the number of tests required for the four independent parameters are 32 cases, shown in Table 2, to match the swelling factor response.

The four parameters used in the experiment are pressure, temperature, initial oil volume, and equilibrium time (see Table 1). The response to the experiments is the swelling factor. According to the parameters and the response, D-Optimal is used as the design model. The total experimental design recommended for the swelling test from D-Optimal is 32 runs as seen in Table 2. The D-Optimal experimental design and the assignment of logical values of parameters results in swelling factors ranging from 1.020 to 1.290. Attempts of extrapolation are done in the analysis based on this range of values and on logical understanding of the behavior of relationship between the swelling factor and its affecting parameters so that it minimizes erroneous observation.

Figure 1 shows the flow diagram of the study. An oil sample is taken from a reservoir within the Air Benakat Formation located in Jambi Province, Indonesia. The oil properties and composition are shown in Tables 3a and 3b, respectively. Since we used only one oil sample, the discussion of the effect of composition on the optimum initial oil volume and equilibrium time will be beyond the scope and objectives of the present study. However, it is logically believed that the amount of hydrocarbon

Table 2. D-Optimal experimental design and results.

No. experiment	Pressure, psi	Temperature, F	Initial oil volume, cc	Equilibrium time, min	Swelling factor
1	900	150	.5	30	1.040
2	2100	150	.5	30	1.170
3	2500	145	.5	30	1.196
4	600	140	.5	30	1.073
5	2500	140	.5	30	1.250
6	600	150	1.5	30	1.020
7	1800	140	1.5	30	1.260
8	900	150	3.4	30	1.090
9	2500	150	3.4	30	1.230
10	600	145	3.4	30	1.030
11	600	140	3.4	30	1.060
12	2500	140	3.4	30	1.260
13	2500	150	.5	75	1.210
14	1500	140	.5	75	1.270
15	600	145	1.5	75	1.070
16	1800	150	3.4	75	1.280
17	2500	140	3.4	75	1.260
18	600	150	.5	120	1.080
19	900	150	.5	120	1.120
20	2100	145	.5	120	1.200
21	600	140	.5	120	1.110
22	2500	140	.5	120	1.250
23	2500	150	1.5	120	1.270
24	900	145	1.5	120	1.140
25	2100	140	1.5	120	1.240
26	600	150	3.4	120	1.100
27	2500	150	3.4	120	1.290
28	1800	145	3.4	120	1.230
29	600	140	3.4	120	1.060
30	900	140	3.4	120	1.110
31	2100	140	3.4	120	1.250
32	2500	140	3.4	120	1.250

components that can be extracted by CO₂ as well as the shrinkage phenomena will affect the swelling factor and thus the optimum condition.

The swelling test is conducted using a small-volume high-pressure view cell. The total cell volume is 4 cc. Due to the small volume; the experiment does not use a stir bar inside the cell. The designed initial volumes of oil in the cell and equilibrium times are applied. In this experiment, the initial oil volume ranges from 12.5 to 85% of the cell volume and the equilibrium time ranges from 30 to 120 min.

The first step in our swelling test procedure is to clean up the cell using toluene and dry it using nitrogen. The oil sample is first filtered using a filtration paper of .5 μm in size. As mentioned, the total cell volume used is about 4 cc. The cell is then filled with 3 (three) cases of initial oil sample volumes of .5, 1.5, and 3.4 cc, or about 12.5, 37.5, and 85% of the total cell volume, at the pressure of 14.7 psi. The desired temperature bath is set using the temperature control. In the experiments, three temperature conditions of 140, 145, and 150 °F are applied. The CO₂ is then flowed into the cell using the ISCO pump. The desired pressure is controlled by the pump. The pressure values applied are 600, 900, 1500, 1800, 2100, and 2500 psi. Note again that the stirrer bar is not used due to the small volume of

the cell. The equilibrium conditions applied at 30, 75, and 120 min. When the system has reached its equilibrium condition, the camera starts to take pictures of the fluid level inside the cell. After the experiment is finished, the pressure inside the cell is reduced gradually. Afterward, the cell is cleaned up from the oil sample using toluene and dried by using nitrogen. The swelling test experimental diagram is shown in Figure 2.

Results and discussion

The swelling test experiment is conducted under the DOE. The data acquired from the experiment are used as input to the RSM procedure. In the RSM, the coefficient of the model is calculated by the multiple regression analysis with the quadratic model given by the following equation.

$$\begin{aligned}
 Y = & 1.226 + 0.088\text{Pre} - 0.012\text{Temp} + 0.009\text{Oil} \\
 & + 0.016\text{Equ} - 0.059\text{Pre.Pre} + 0.022\text{Temp.Temp} \\
 & - 0.014\text{Oil.Oil} - 0.025\text{Equ.Equ} + 0.009\text{Pre.Oil} \\
 & - 0.006\text{Pre.Equ} + 0.019\text{Temp.Oil} + 0.018\text{Temp.Equ}
 \end{aligned}
 \quad (1)$$

where the variables Pre, Temp, Equ, and Oil represent pressure, temperature, equilibrium time, and initial oil volume, respectively, as shown in Table 1.

The statistical results of the multiple regression function are evaluated using the analysis of variance (ANOVA) shown in Table 4. It is found that the R² value represents good correlation between the observed and the predicted values. The higher R² (.987) and adjusted R² (.979) also indicate the efficiency of the model suggesting that 98.7% and 97.9% variations could be accounted for by the model equation, respectively. At the same time, the very low coefficient of the residual standard deviation (RSD = .0128) clearly informs a high degree of precision and reliability of the experimental values and is in relation to the power of prediction, Q² = .963.

The student's *t*-test of statistics is designed to evaluate the quantitative effect of the main factors. The regression coefficient values of Equation (1) are listed with standard errors and *p*-values in Table 5. The *p*-value is used to check the significance of each coefficient which in turn may indicate the pattern of interaction between variables. Variables of Equ (equilibrium time) and Oil (initial oil volume) have effects on the swelling factor. Those variables are having a small *p*-value (*p* < .05). It is noteworthy that the positive sign indicates a synergistic effect.

Effect of parameters on the swelling factor

The effects of the main parameters and the interaction among the parameters on the swelling factor (SF) are shown in Figure 3. The figure is divided into two

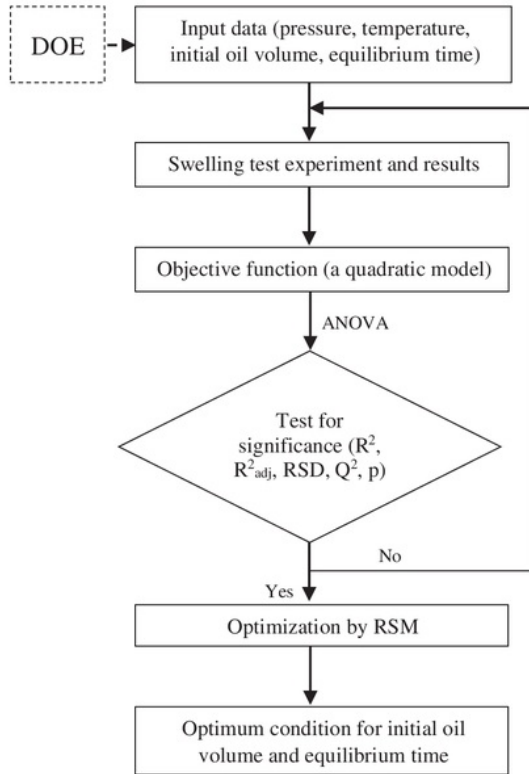


Figure 1. Workflow diagram for the present study.

Table 3a. Sample properties.

Properties	AB-4
API gravity	41.38
Reservoir temperature (T), °F	150
Reservoir pressure (p), psi	1370
Bubble point pressure (p_b), psi	670
Viscosity, c_p	.487

Table 3b. Sample composition.

Component	Symbol	Mole percent	Weight percent
Hydrogen sulfide	H ₂ S	.00	.00
Carbon dioxide	CO ₂	.12	.05
Nitrogen	N ₂	.65	.17
Methane	C ₁	18.50	2.71
Ethane	C ₂	1.79	.59
Propane	C ₃	1.87	.75
Iso-Butane	i-C ₄	.84	.45
n-Butane	n-C ₄	1.37	.73
Iso-Pentane	i-C ₅	1.64	1.08
n-Pentane	n-C ₅	.99	.65
Hexanes	C ₆	2.58	2.03
Heptane plus	C ₇₊	69.65	90.89
		100.00	100.00
Properties of heptane plus			
Specific gravity @ 60/60 °F		.8308	
Molecular weight		142.73	

regions. The first region is that above the zero; this is for the positive coefficients of individual and combined parameters such as Pre, Temp.Temp, Temp.Oil, Temp. Equ, Equ, Oil, and Pre.Oil. The second region is that below the zero; this is for the negative coefficients such as Pre.Pre, Equ.Equ, Oil.Oil, Temp, and Pre.Equ. The figure is of importance to explain the significance of the effect of parameters on the overall outcome of the swelling factor. From the figure, it is easy to recognize that the effects of oil volume and equilibrium time are positive on the swelling factor. This means the incremental value from the parameters leads to the increase of the swelling factor.

Based on the nonlinear results shown in the sensitivity chart, the maximum oil volume, i.e., 3.4 cc or 85% of the total cell volume, is required to get the maximum swelling factor as can be seen on Figure 4a. Note that this condition is achieved when the initial oil nearly fill up the entire cell volume suggesting that further increase in oil volumes may result in a very small increment in the swelling factor. Meanwhile, an optimum condition for the equilibrium time is found to be in between 95 and 105 min as shown by Figure 4b. Considering the maximum swelling factor can be achieved when the initial oil volume of 85% as suggested by Figure 4a, the maximum initial oil volume is clearly greater than those suggested by previous studies (30% as suggested by Harmon and Grigg (1998); 30–37% as suggested by Hand & Pinczewski (1990); 15–53.8% as suggested by Tsau et al. (2010); and 9% as suggested by Wang, 1986). Because of high initial oil volume, more gas is expected to be dissolved into the oil. This phenomenon causes only the oil to swell while the oil shrinking is not likely

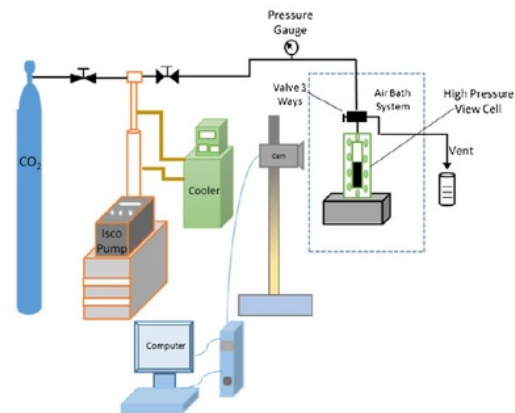


Figure 2. Swelling test experimental diagram.

Table 4. ANOVA for the RSM.

Swelling factor	DF	SS	MS	F	p	SD
Total	32	44.117	1.379	–	–	–
Constant	1	43.873	43.873	–	–	–
Total Corrected	31	.244	.008	–	–	.089
Regression	12	.241	.020	122.73	0	.142
Residual	19	.003	.000			.013
N = 32		Q ² = .963	R ² Adj. = .979		Conf. Lev = .95	
DF = 19		R ² = .987	RSD = .0128			

Table 5. Estimated regression coefficients for the swelling test using coded units.

Swelling factor	Estimate	Standard error	p-value
Constant	1.226	.009	1.49E-29
Pre	.088	.003	5.03E-18
Temp	-.012	.003	1.82E-04
Oil	.009	.003	1.98E-03
Equ	.016	.003	3.72E-06
Pre.Pre	-.059	.006	2.73E-08
Temp.Temp	.022	.006	1.95E-03
Oil.Oil	-.014	.007	5.91E-02
Equ.Equ	-.025	.006	8.23E-04
Pre.Oil	.009	.003	9.56E-03
Pre.Equ	-.006	.003	6.23E-02
Temp.Oil	.019	.003	1.90E-06
Temp.Equ	.018	.003	4.52E-06

to occur. Furthermore, the optimum equilibrium time is found to be longer than 30 to 60 min, i.e., the equilibrium time suggested by Tsau et al. (2010), as can be seen in Figure 4b. The longer contact time makes the CO₂ to be more soluble in the oil and, therefore, the oil swelling becomes larger. However, at a certain time, as the CO₂ solubility reaches its maximum condition, the oil swelling starts to be smaller. The absence of a stirrer bar may be the second factor expected as another reason why the equilibrium time is longer than that in previous

studies. Without a stirrer bar used in the view cell, the CO₂ will require some more time to be soluble in the oil. With the condition, it is found that the increment of the equilibrium time from 30 min to the optimum condition leads to the increase of the swelling factor to approximately 4.9%. Meanwhile, the increase of the oil volume of .5 cc or 12.5% to its maximum condition increases the swelling factor to approximately 6.7%.

Optimization of parameters by the RSM

The RSM is used to identify the optimum and maximum values of the independent parameters at which the dependent parameters arrive as the maximum response. The 2D contour and 3D response surface plots represent the prediction of the dependent parameters for a set of independent parameters. Figures 5a and 5b show the contour plot and the response surface plot, respectively. Note that the axis range values are selected in order to show the optimal condition while it is still within the range of our experimental design results. The value of the maximum of the surface plot is confined in the smallest ellipse in the contour diagram. Elliptical contours are generally obtained when perfect interaction between the independent parameters occurs. The value of independent parameters in the smallest contour area and the corresponding maximum are determined to be the optimum condition for the response parameters.

The contour and the response surface plots suggested that the best oil volume is 3.4 cc or 85% of the total cell volume and the equilibrium time is 101 min. The parameters lie in the smallest region in Figures 5a and 5b where the maximum swelling factor reaches the value of 1.299.

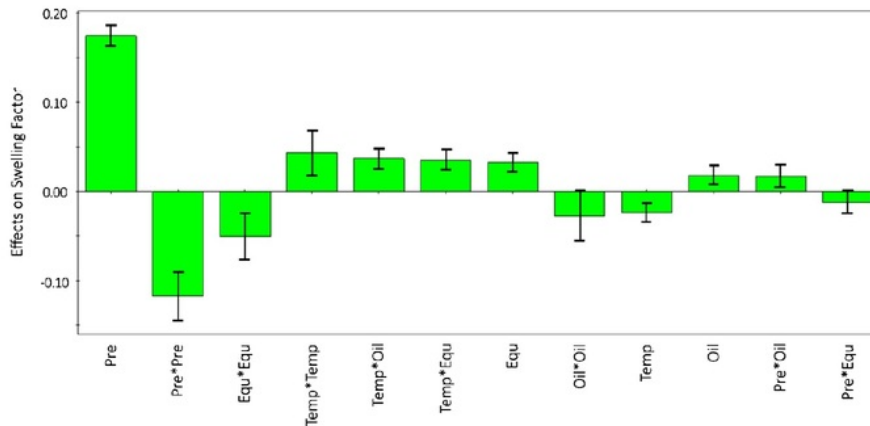


Figure 3. Ranking of factors affecting the SF.

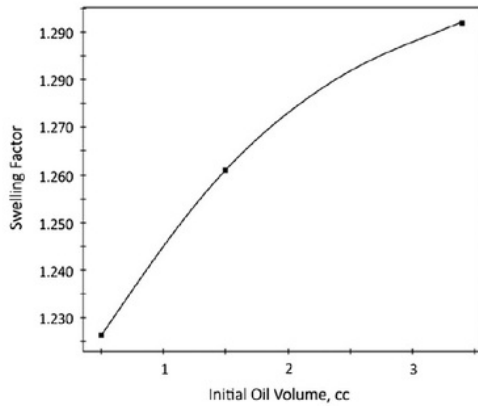


Figure 4a. SF vs. oil volume.

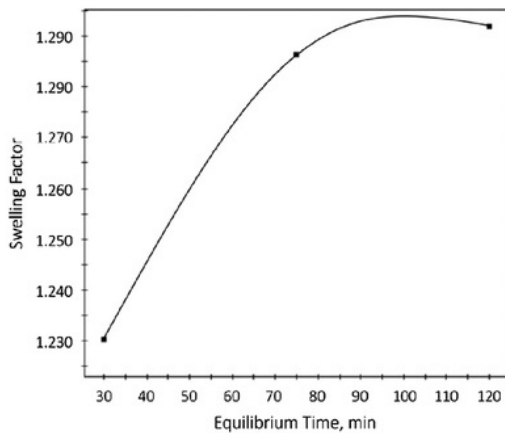


Figure 4b. SF vs. equilibrium time.

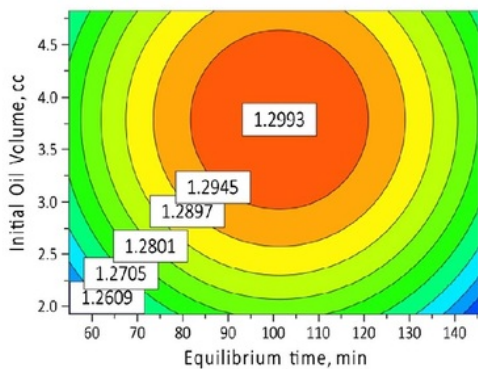


Figure 5a. Contour plot showing optimal condition.

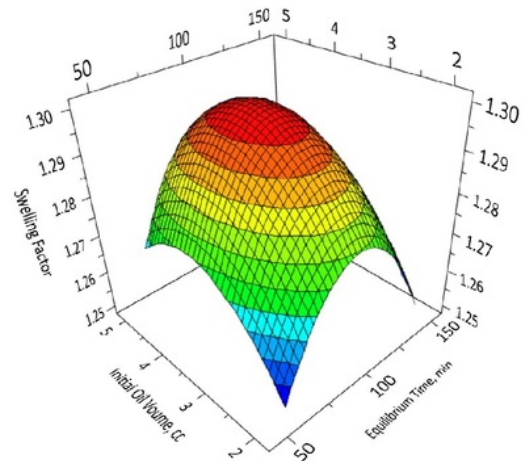


Figure 5b. Response surface plot showing optimal condition.

Conclusions

Based on the results of the present study, valuable information and conclusions can be drawn as follows:

- (1) The RSM has been proved to be appropriate for establishing the optimum parameters to yield the maximum swelling factor.
- (2) The RSM reduces the time consumed in the experimental study by providing the number of experiments to obtain the effective DOE.
- (3) The initial oil volume and the equilibrium time may affect the value of swelling factor considerably.
- (4) The increase of CO_2 solubility in the oil prolongs the equilibrium time and increases the swelling factor. However, at a certain time, the swelling factor decreases due to the oil shrinkage.
- (5) Higher initial oil volume may have to be used in the cell for taking the advantage of oil swelling characteristic. At this condition, the oil shrinkage does not occur promoting the increase of swelling factor.

Disclosure statement

No potential conflict of interest was reported by the authors.

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