Petroleum Research 6 (2021) 264–270

Contents lists available at ScienceDirect

Petroleum Research

journal homepage: http://www.keaipublishing.com/en/journals/petroleum-research/

# Estimation of pore volumes to breakthrough number in limestone cores by derivation of an empirical model

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A R T I C L E I N F O

*Article history:*
Received 17 December 2020
Received in revised form
11 February 2021
Accepted 21 February 2021
Available online 25 February 2021

*Keywords:*
Acidizing
Pore volumes to breakthrough
Limestone
Wormhole
Empirical model

A B S T R A C T

Acidizing in carbonate formations is an inevitable stimulation treatment method for oil and gas wells. In the limestone, acidizing stimulation makes capillary wormholes to increase fluids flow reservoir production. The pore volume to breakthrough number is one of the main indexes for recognizing the wormhole structure. Therefore, finding the pore volume to breakthrough number is one of the main goals in the limestone acidizing. Obtaining this number is always required for experimental works, which needs time, energy and cost. The purpose of this research is to develop an empirical method to estimate an acceptable result for this number merely by implementing limestone core and acid properties without any experimental work. In order to create a wormhole, an empirical method is developed using the law of conservation of mass considering that the core of limestone as an isolated package and the overall mass is constant in this package in the acidizing period. Also, to develop the mathematical section, the Damköhler number is used. Since this number must be calculated experimentally, a constant number is created in the model to eliminate the Damköhler number. An average accuracy of 92.31% is obtained for the developed empirical model by comparing the results obtained from the other three experimental and numerical works. This study conclusively provides a thoroughly empirical method for estimating a high accuracy of the pore volume to breakthrough number by only using known physical properties limestone core and acid.



## 1. Introduction

This empirical modelling involves estimating the scale of the acid injection flow rate through the wormholes formed inside the limestone core that is called pore volume to breakthrough number ($PV_{BT}$) based on mass balance law (Volkenstein, 2009). The compressibility of pore volumes in the cores is related to the pore aspect ratio, porosity, and the proportion of pore volumes (Sui et al., 2020). The empirical method maintains the relative of mass transport and chemical reaction between limestone core and injected acid. Grounded on this technique, during the acidizing process and wormhole creation, the overall mass in the limestone core as a closed system is considered to be constant. Also, to determine the $PV_{BT}$, the shape of the wormhole is considered to be a cylindrical capillary tube. According to mass balance law, the injected acid to the limestone core as an input mass minus the product fluid came out of it as an output mass shows the created wormhole volume inside the core as a change of masses. To develop the mathematical section, the Damköhler number ($N_{Da}$) is used (Fredd, 2000; Fredd & Fogler, 1998a, 1998b, 1998c, 1999; Fredd et al., 1997; Gdanski, 1999; Talbot and Gdanski, 2008). This dimensionless number is defined as the time of fluid flow to the time of chemical reaction (Fogler, 1999). Since experimental works must calculate this number, the rate of core dissolution is defined in the model. This factor shows the wormhole growing rate in the core (Inger, 2001). Also, a coefficient constant number which is specific for each type of limestone core and acid is added to the model to eliminate the $N_{Da}$. The added coefficient number has a linear relative with the $N_{Da}$.

To enhance oil recovery technologies by using chemical fluids, the characterization of reservoirs is an important aspect (Fang et al., 2018; Qi et al., 2018; Sun et al., 2018). The optimum acid injection

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https://doi.org/10.1016/j.ptlrs.2021.02.002

rate depends on the acid concentration, the core properties, and the temperature; and among all, rock properties have the most influence on the optimum acid rate. Comparing to limestone, in slow dolomite reactions, a significant rise in acid mass and a meaningful decrease in rate are required. Also, the temperature rise increases the reaction rate of dolomite. Therefore, the results of the acid injection in dolomite at high temperatures are aligned with the acid injection in limestone (Akanni & Nasr-El-Din, 2015; Dong, 2018; Dong et al., 2016; Economides and Nolte, 1989; ; Glasbergen et al., 2005; Glasbergen et al., 2009a,b; Liu et al., 2012; Mahmoud, Nasr-El-Din, De Wolf and LePage, 2010; Sidaoui et al., 2018).

Concentration changes by using chemical balance equation rules in the limestone core and acid during acidizing are used for the chemical section of the empirical model. The injected acid cannot solve the entire limestone core since the weight mass of injected acid is less than weight masses of limestone core this phenomenon known as limiting reagent law. "The limiting reagent or limiting reactant in a chemical reaction is the substance that is totally consumed when the chemical reaction is complete" (Zumdahl, 1995).

The constant equations for different kinds of acids such as mineral, organic, complex are used for derivation of the empirical model. The final equation for the model that can calculate the $PV_{BT}$ number contains only physical limestone core and injected acid properties, and this empirical model can estimate the $PV_{BT}$ number with acceptable results without any experimental works. Using the same method for complex acids and limestone cores shows the accuracy of 81.36% and, the accuracy of 95.98% for hydrochloric acid and carbonate cores (Lohrasb & Junin, 2019a, 2019b). On the other hand, an empirical model that can estimate this number has not yet been developed for hydrochloric acid and limestone cores. Therefore, in this study, an empirical model capable of estimating $PV_{BT}$ based on the properties of acids and carbonate formations without any experimental work was developed.

## 2. Methodology

Three categories of experimental, semiempirical and numerical works were used to produce and test the model for the data collected in this empirical model. The first category is experimental data and was collected from Fredd and Fogler (1998b) work. A single type of Indiana limestone core and five varieties of acids have been collected. Indiana limestone with an average porosity of 20%, a diameter of 3.81 cm and a length of 10.16 cm. Regarding the injected acids, hydrochloric acid (*HCl*) with a concentration of 0.5 M, acetic acid (*HAc*) with a concentration of 0.5 M, Ethylenediaminetetraacetic acid (*EDTA*) with a concentration of 0.25 M, Cyclohexylenedinitrilotetraacetic acid (*CDTA*) with a concentration of 0.25 M, and Diethylenetriaminepentaacetic acid (*DTPA*) with a concentration of 0.25 M was used. The second category is semi-empirical data is one type of limestone core and one type of acid with two different temperatures and collected from Buijse and Glasbergen (2005), the core is Indiana limestone with a porosity of 20%, diameter of 5.08 cm, and length of 20 cm and the injected acid is *HCl* with a concentration of 1.5 M in 25 °C and 60 °C. Furthermore, for the third category, one type of limestone core and one type of acid were obtained from Maheshwari and Balakotaiah (2013); limestone with a porosity of 20%, diameter of 3.81 cm, and length of 10.16 cm. The injected acid is *HCl*, with a concentration of 0.5 was used.

For data processing, the method is focused on the maintenance of mass law for the empirical part of the model, and the entire core was defined to be a close system. Therefore, the total mass in the process should be constant throughout the process of fluid injection into the core and wormhole creation. Also, it is presumed that the wormhole has a cylinder shape so that its volume can be measured. By using this method, the mass entry at the one side minus output mass at the other side of carbonate core as a system shows the occurred changes in the system that creates the volume of the wormhole. The dimensionless Damköhler number ($N_{Da}$) is used for the mathematical section of model development. This number is defined as the flow time to chemical time scale ratio. A coefficient constant was utilized in this empirical model to eliminate $N_{Da}$ number, which is basically calculated experimentally.

## 3. Model development

For model development, the wormhole is considered as a capillary tube with the cross section of $a$ with length of $l$ and diameter of $d$ created with acid injection flow rate of $q_1$ and concentration of $C_1$ inside the limestone core with the length of $X$ and cross section of $A$. The initial equation based on the mass balance law is presented in Fig. 1 and Equation (1) and the initial equation is expanded step by step as below.

The empirical model is initiated by expanding the Equation (1) using the following known Equations (3)–(5) to determine $PV_{BT}$ number.

$$C_1 = \frac{m_a}{V_i} \tag{3}$$

$$q_a = \frac{\pi d l k}{N_{Da}} \tag{4}$$

$$V_w = \frac{\pi d^2 l}{4} \tag{5}$$

In the mentioned equations, $m_a$ is the mass weight of injected acid, $V_i$ is the volume of injected acid, $q_a$ is acid injected rate presented by Fredd and Fogler (1998b), $\pi$ is equal to 3.14, $k$ is acid and core dissolution rate constant and $V_w$ is wormhole volume. The limestone core and acid reaction time or time differential ($\Delta t$) is the

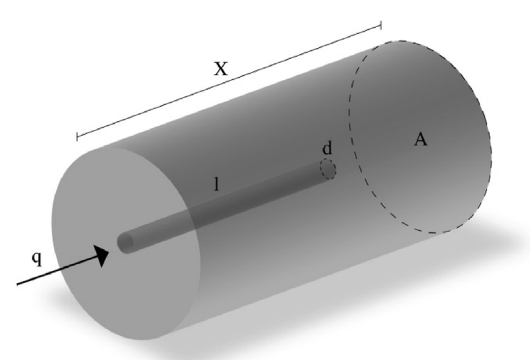


**Fig. 1.** Capillary tube wormhole created inside a limestone core.

$$\left\{ \begin{matrix} \text{mass of acid} \\ \text{into the core} \end{matrix} \right\} - \left\{ \begin{matrix} \text{mass of produt fluid} \\ \text{out of the core} \end{matrix} \right\} = \left\{ \begin{matrix} \text{accumulation and rate of} \\ \text{chages of mass inside the core} \end{matrix} \right\} \tag{1}$$

$$\frac{\{q_1 C_1\}_{in} - \{q_2 C_2\}_{out}}{\Delta C} = \frac{al}{\Delta t} \tag{2}$$

same as wormhole creation time that is considered as $T$ in this model. Therefore, the empirical model is developed by the definition of $PV_{BT}$ number. "The number of pore volumes to break through is defined as the ratio of the volume of fluid injected to achieve channel breakthrough to the volume of the pore space in the core" (Fredd and Fogler, 1999). Assuming that the injected acid flow will be stopped somewhere in the middle of the limestone core and, on the other side of the core there is no fluid flow rate and $q_2$ is equal to zero, the initial equation is derived step by step as Equations (6)–(10).

$$\frac{q_1C_1 - q_2C_2}{\Delta C} = \frac{al}{\Delta t} \tag{6}$$

$$\frac{q_1C_1 - q_2C_2}{\Delta C} = \frac{\pi d^2 l}{4T} \tag{7}$$

$$q\frac{m_a}{V_i} = \frac{\pi d^2 l \Delta C}{4T} \tag{8}$$

$$\frac{V_c Ø}{V_i} = \frac{\pi d^2 l \Delta C\ V_c\ Ø}{4\ q\ m_a\ T} \tag{9}$$

$$PV_{BT} = \frac{4\ q\ m_a\ T}{\pi\ d^2\ l\ \Delta C\ V_c\ Ø} \tag{10}$$

Where $C_2$ is product fluid concentration, $\Delta C$ is fluid concentration changes, $V_c$ is limestone core volume and $Ø$ is the porosity of the core, the dissolution rate ($D_R$) and $N_{Da}$ equations is used to expand the $PV_{BT}$ number equation as below.

$$\pi dlk = \frac{V_w}{T} = D_R \tag{11}$$

$$q = \frac{V_w/T}{N_{Da}} = \frac{D_R}{N_{Da}} \tag{12}$$

$$PV_{BT} = \frac{q\ m_a\ T}{V_w\ V_c\ \Delta C\ Ø} \tag{13}$$

$$PV_{BT} = \frac{m_a}{N_{Da}\ V_c \Delta C\ Ø} \tag{14}$$

According to the objective of this study, the main purpose is finding the $PV_{BT}$ number without any experimental work and just by physical properties of limestone core and acid. But as shown in Equation (14), the $N_{Da}$ number must be calculated experimentally, and concentration changes is not known. To find the concentration changes, the chemical balance equation law was used. For example, the mass concentration of product fluid in the reaction of hydrochloric acid ($HCl$) with the molarity of 0.5 mol per liter and a limestone core was calculated and shown in Table 1 and the concentration changes could be calculated by Equations (16) and (17).

$$2HCl + CaCO_3 \rightarrow CaCl_2 + CO_2 + H_2O \tag{15}$$

$$C_2 = \frac{\sum m}{V_i} \tag{16}$$

$$\Delta C = C_2 - C_1 \tag{17}$$

Now the only parameter that still needs to be calculated experimentally is $N_{Da}$ number. To solve this problem, $N_{Da}$ numbers from Fredd and Fogler (1998b) experimental work were used in the Equation (14) (primary equation). Equation 18, and 19 shows the chemical reaction of Acetic acid ($HAc$), and Ethylenediaminetetra-acetic acid ($EDTA$) with limestone cores; and Fig. 2 shows the calculated $PV_{BT}$ based on primary equation and Fredd and Fogler (1998b) experimental results for these three acids ($HCl$, $HAc$, $EDTA$). Where the vertical axis shows $PV_{BT}$ and the horizontal axis shows acid injection rate ($cm^3/min$).

$$2CH_3COOH + CaCO_3 \rightarrow Ca(CH_3CO_2)_2 + CO_2 + H_2O \tag{18}$$

$$C_{10}H_{16}N_2O_8 + CaCO_3 \rightarrow CaC_{10}H_{16}N_2O_8 + CO_3 \tag{19}$$

As it is clear in the Fig. 2, the shape of curves in the empirical model and experimental results are almost the same but they have a huge deviation. To eliminate this deviation and calibrate Equation (14), a specific dimensionless coefficient number ($Co.$) for each $PV_{BT}$ number was multiplied to the empirical model equation as shown in Table 2, and by analyzing the results; it was found that the $Co.$ have a linear and direct relation with $N_{Da}$ for each acid as shown in Fig. 3.

$$PV_{BT} = \frac{Co.\ m_a}{N_{Da}\ V_c\ \Delta C\ Ø} \tag{20}$$

Therefore, the main empirical equation of $PV_{BT}$ for mentioned acids were presented by Equations (21)–(23). And the developed empirical model was evaluated with others' works as below.

$$\text{For } HCl \rightarrow Co. = 1.3791 N_{Da} \rightarrow PV_{BT} = \frac{1.3791\ m_a}{V_c\ \Delta C\ Ø} \tag{21}$$

$$\text{For } HAc \rightarrow Co. = 0.8333 N_{Da} \rightarrow PV_{BT} = \frac{0.8333\ m_a}{V_c\ \Delta C\ Ø} \tag{22}$$

$$\text{For } EDTA \rightarrow Co. = 0.3454 N_{Da} \rightarrow PV_{BT} = \frac{0.3454\ m_a}{V_c\ \Delta C\ Ø} \tag{23}$$

## 4. Evaluation of empirical model equations

To evaluate the accuracy and deviation of the empirical model equations, calculated results were compared to the $PV_{BT}$ results presented by Fredd and Fogler (1998b), Buijse and Glasbergen (2005), and Maheshwari and Balakotaiah (2013) (as reviewed in Table 3); and evaluation of empirical model were measured by three statistic methods including Standard Deviation ($SD$), Mean

**Table 1**
Mass concentration for limestone core and 0.5 M *HCl*.

| | Constant | Compound | Molar concentration (*mol/lit*) | Molar mass (*g/mol*) | Weight Concentration (*g/lit*) | Mass Concentration (*g/cm³*) |
|---|---|---|---|---|---|---|
| *Injected acid* | 2 | $HCl$ | 0.5 | 36.46094 | 18.2305 | 0.0182 |
| *Product fluid* | 1 | $CaCl_2$ | 0.25 | 110.984 | 27.746 | 0.0277 |
| | 1 | $CO_2$ | 0.25 | 44.0095 | 11.0024 | 0.011 |
| | 1 | $H_2O$ | 0.25 | 18.01528 | 4.5038 | 0.0045 |

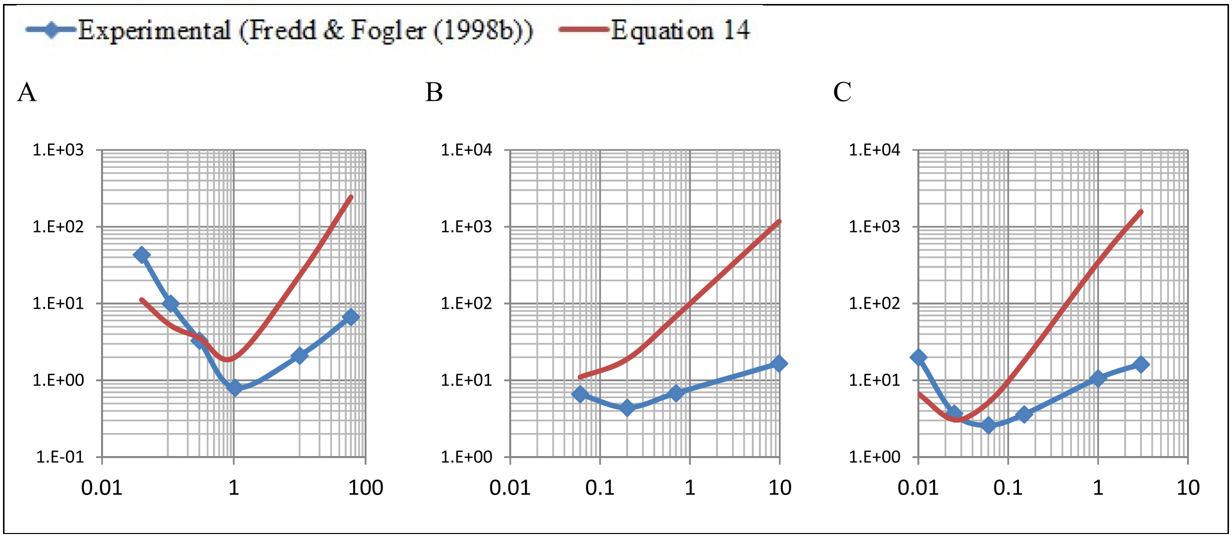


**Fig. 2.** Comparison of the results obtained by Equation (14) and experimental results given by Fredd and Fogler (1998b) for three acids and limestone cores. A) 0.5 M *HCl*; B) 0.5 M *HAc*; C) 0.25 M *EDTA*.

**Table 2**
Calculated coefficient numbers by Equation 15.

| 0.5 M HCl | | | 0.5 M HAc | | | 0.25 M EDTA | | |
|---|---|---|---|---|---|---|---|---|
| $q$ ($cm^3/min$) | $N_{Da}$ | Co. | $q$ ($cm^3/min$) | $N_{Da}$ | Co. | $q$ ($cm^3/min$) | $N_{Da}$ | Co. |
| 0.04 | 2.8 | 3.8615 | 0.06 | 0.72 | 0.6000 | 0.01 | 8.7 | 3.0027 |
| 0.11 | 1.4 | 1.9308 | 0.2 | 0.28 | 0.2333 | 0.025 | 3.5 | 1.2066 |
| 0.3 | 0.67 | 0.9240 | 0.7 | 0.12 | 0.1000 | 0.06 | 1.5 | 0.5056 |
| 1.05 | 0.29 | 0.3999 | 9.8 | 0.17 | 0.0142 | 0.15 | 0.6 | 0.2031 |
| 10 | 0.066 | 0.0910 | | | | 1 | 0.09 | 0.0308 |
| 60 | 0.02 | 0.0276 | | | | 3 | 0.03 | 0.0103 |

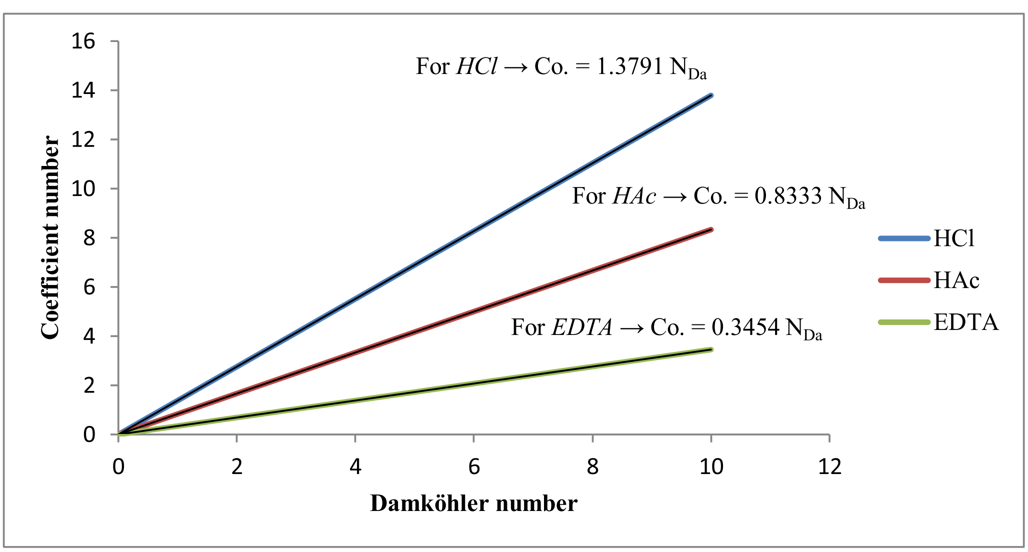


**Fig. 3.** Coefficient number and Damköhler number relation.

**Table 3**
Overview of the others works.

| Author | Acid | Acid Molarity (*mol/lit*) | Core | Core porosity (%) | Core length (*cm*) | Core diameter (*cm*) |
|---|---|---|---|---|---|---|
| Fredd and Fogler (1998b) | *HCl* | 0.5 | limestone | 20 | 10.16 | 3.81 |
| | *HAc* | 0.5 | | | | |
| | *EDTA* | 0.25 | | | | |
| | *CDTA* | 0.25 | | | | |
| | *DTPA* | 0.25 | | | | |
| Buijse and Glasbergen (2005) | *HCl, 25°C* | 1.5 | limestone | 20 | 20 | 5.08 |
| | *HCl, 60°C* | 1.5 | | | | |
| Maheshwari and Balakotaiah (2013) | *HCl* | 0.5 | limestone | 20 | 10.16 | 3.81 |

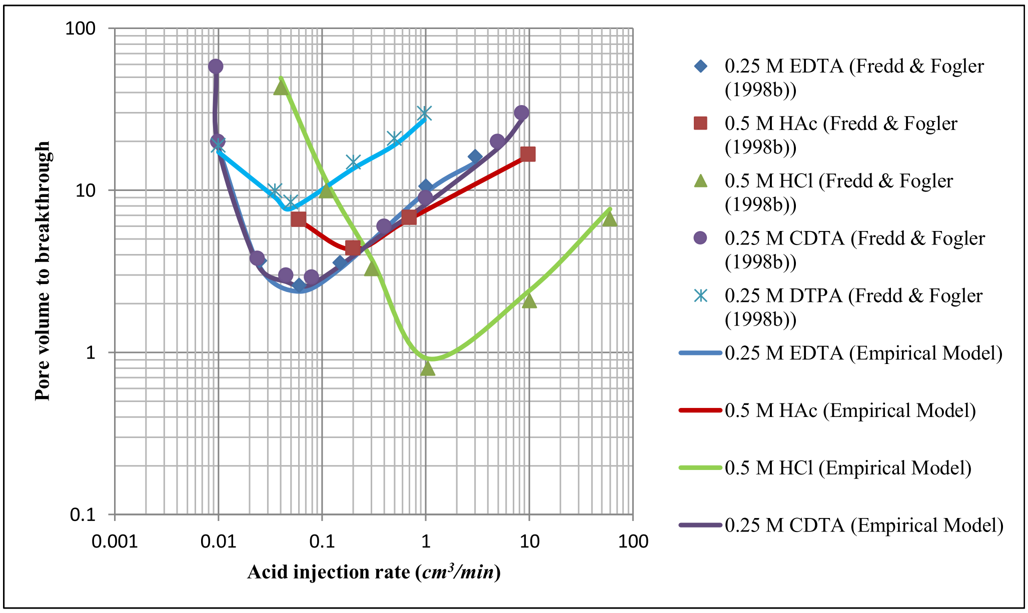


**Fig. 4.** Evaluation of the empirical model with Fredd and Fogler (1998b) results.

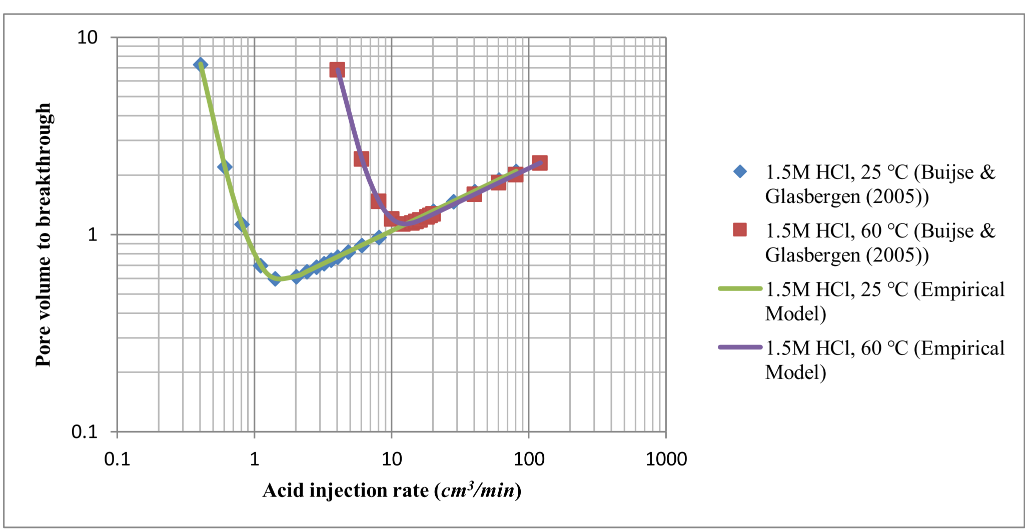


**Fig. 5.** Evaluation of the empirical model with Buijse and Glasbergen (2005) results.

Absolute Percentage Error (*MAPE*), and Accuracy percentage (Equation (24)–26).

$$SD = \sqrt{\frac{\sum_{i=1}^{N}(PV_{actual} - PV_{model})^2}{N}} \tag{24}$$

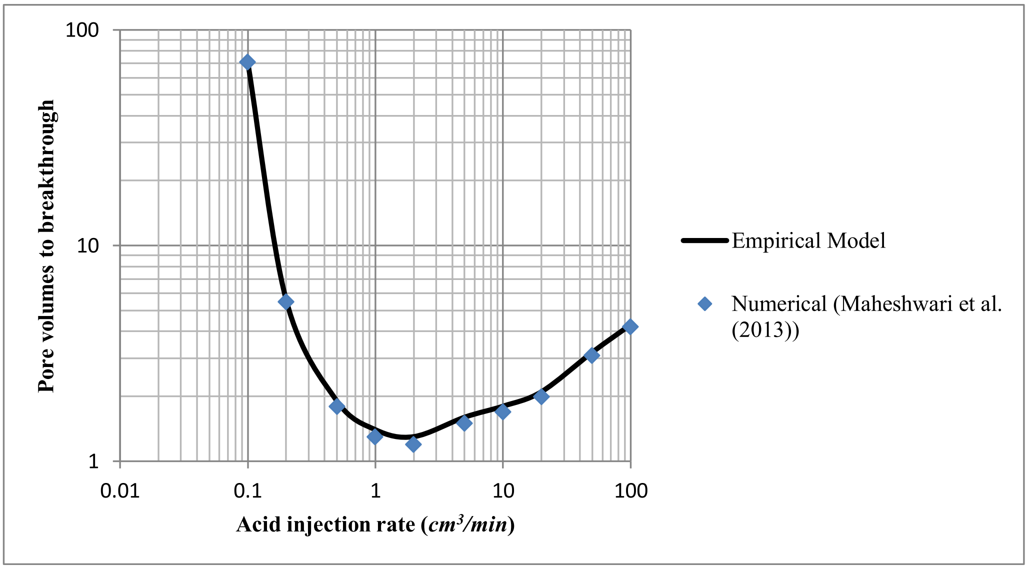


**Fig. 6.** Evaluation of the empirical model with Maheshwari and Balakotaiah (2013) results.

**Table 4**
Evaluation of the model.

| | Acid type | SD | MAPE (%) | Accuracy (%) |
|---|---|---|---|---|
| Fredd and Fogler (1998b) | *0.5 M HCl* | 0.0003 | 0.01 | 99.99 |
| | *0.5 M HAc* | 0.0004 | 0.02 | 99.98 |
| | *0.25 M EDTA* | 0.0597 | 0.99 | 99.01 |
| | *0.25 M CDTA* | 2.8159 | 19.65 | 80.35 |
| | *0.25 M DTPA* | 6.5973 | 35.27 | 64.73 |
| Buijse and Glasbergen (2005) | *1.5 M HCl, 25°C* | 0.1789 | 0.45 | 99.55 |
| | *1.5 M HCl, 60°C* | 0.2059 | 0.45 | 99.55 |
| Maheshwari and Balakotaiah (2013) | *0.5 M HCl* | 0.0999 | 4.67 | 95.33 |
| Average | | 1.24 | 7.69 | 92.31 |

$$MAPE = \frac{1}{N}\frac{\sum_{i=1}^{N}|PV_{actual} - PV_{model}|}{PV_{actual}} \times 100 \tag{25}$$

$$\%\,Accuracy = 100 - MAPE \tag{26}$$

Fig. 4–6 shows the evaluation of the developed empirical model with three different other works.

For the verification, the developed empirical model was evaluated by some statistics methods. Table 4 shows the calculated *SD*, *MAPE* and Accuracy percentage for all 76 samples from three different works were used in this paper.

## 5. Conclusions

The average accuracy for the developed empirical model has been obtained by evaluating the results with 76 samples from three different works, and it is shown to be 92.31% accuracy. Statistical comparison of the first classification of experimental results with empirical results indicates that mineral and organic acids had better results than complex acids in the empirical model. Evaluation of the second classification of the experimental results with empirical model results shows that temperature increase of mineral acid injection inside limestone core has no meaningful effect on the accuracy percentage of the developed empirical model, while the *SD* shows better results in higher temperatures. Also, a comparison of the third classification proves that higher acid concentration leads to more accurate results for hydrochloric acid.

## Acknowledgment

The authors would like to thank Universiti Teknologi Malaysia (UTM) for providing support.

## Symbols and Abbreviations

| | |
|---|---|
| $\Delta C$ | Fluid concentration changes ($gr/cm^3$) |
| $A$ | Cross section of the core ($cm^2$) |
| $a$ | Cross section of the wormhole ($cm^2$) |
| $C_1$ | Acid concentration before injection ($gr/cm^3$) |
| $C_2$ | Fluid concentration after reaction ($gr/cm^3$) |
| $CaCO_3$ | Limestone (*Calcium Carbonate*) |
| *CDTA* | Cyclohexylenedinitrilotetraacetic acid ($C_{14}H_{22}N_2O_8$) |
| *Co* | Coefficient number (*Dimensionless*) |
| $d$ | Wormhole diameter ($cm$) |
| $D_R$ | Dissolution rate constant ($cm^3/min$) |
| *DTPA* | Diethylenetriaminepentaacetic acid ($C_{14}H_{23}N_3O_{10}$) |
| *EDTA* | Ethylenediaminetetraacetic acid ($C_{10}H_{16}N_2O_8$) |
| *HAc* | Acetic acid ($CH_3COOH$) |
| *HCl* | Hydrochloric acid (*HCl*) |
| $k$ | Overall dissolution rate ($cm/min$) |
| $l$ | Wormhole length ($cm$) |
| $M$ | Molarity ($mol/lit$) |
| $m_a$ | Mass of acid ($gr$) |
| *MAPE* | Mean absolute percentage error (%) |
| $N$ | Number of samples |
| $N_{Da}$ | Damköhler number (*Dimensionless*) |

| | |
|---|---|
| Ø | Porosity |
| $PV_{BT}$ | Pore volume to breakthrough (*Dimensionless*) |
| $q$ | injection rate ($cm^3/min$) |
| $SD$ | Standard deviation |
| $T$ | Processing time ($min$) |
| $t$ | Time ($min$) |
| $V_c$ | Core volume ($cm^3$) |
| $V_i$ | Volume of injected fluid ($cm^3$) |
| $V_w$ | Wormhole volume ($cm^3$) |
| $X$ | length of core ($cm$) |
| $\pi$ | Pi number (*3.14*) |

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