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## Article

# Study of the kinetics of carboxymethyl cellulose synthesis in a screw reactor

Technology audit and production reserves

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*Reference:* Konovalenko, Kateryna/Beznosyk, Yurii et. al. (2021). Study of the kinetics of carboxymethyl cellulose synthesis in a screw reactor. In: Technology audit and production reserves 6 (3/62), S. 15 - 20.

<http://journals.uran.ua/tarp/article/download/244777/243992/570477>.

doi:10.15587/2706-5448.2021.244777.

This Version is available at:

<http://hdl.handle.net/11159/7236>

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## STUDY OF THE KINETICS OF CARBOXYMETHYL CELLULOSE SYNTHESIS IN A SCREW REACTOR

*The object of research is the reactor for the synthesis of carboxymethyl cellulose. An important indicator of the quality of sodium carboxymethyl cellulose, which determines the field of its application, is the degree of polymerization. However, obtaining a product with a specific parameter under industrial conditions is associated with a number of difficulties. Therefore, important research tasks are the development of a mathematical model of the kinetics of carboxymethyl cellulose synthesis, experimental studies to determine the rate constants of synthesis reactions, modeling of a screw reactor for the synthesis of carboxymethyl cellulose, and computer studies.*

*When studying the kinetics of reactions of carboxymethyl cellulose, one of the possible approaches was to use a quasi-homogeneous model, which is widely used in modeling processes on a catalyst grain. This approach is used to describe and analyze individual stages; however, a number of difficulties arise in heterogeneous reactions of cellulose. In the course of these reactions, the properties of the solid phase change and the processes, respectively, are unsteady in time. The reaction does not take place on the surface of hard particles, but in the entire volume of the fibers. The concentration and reactivity of cellulose hydroxides, water, and products formed during the reaction remain approximately constant; therefore, the use of a quasi-homogeneous model is quite acceptable and does not cause additional mathematical difficulties. As a result of these experiments, according to the obtained integral curves, the method of least squares was used to find the constants. To determine the values of the kinetic constants, an experiment was carried out in an integral isothermal reactor. During the experiments, the degree of substitution of carboxymethyl cellulose and the concentration of free alkali were measured. As a result of numerous implementations of the search task, the values of the constants and activation energies were obtained. This kinetic modeling approach can be used in the synthesis of other cellulose ethers. The rate constant of the synthesis reaction depends on the process conditions. Using the proposed approach to describing the interaction of cellulose with a reagent, the reaction mixture considered as a quasi-homogeneous system can be described using a single-phase flow model.*

**Keywords:** cellulose ethers, solid-phase production method, tubular screw reactor, plug-flow mode.

Received date: 21.06.2021

Accepted date: 11.08.2021

Published date: 08.12.2021

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### How to cite

Konovalenko, K., Beznosyk, Y., Bugaieva, L. (2021). Study of the kinetics of carboxymethyl cellulose synthesis in a screw reactor. *Technology Audit and Production Reserves*, 6 (3 (62)), 15–20. doi: <http://doi.org/10.15587/2706-5448.2021.244777>

### 1. Introduction

Carboxymethyl cellulose (CMC) is a cellulose ether. It appears in the form of sodium salts with the assistance of alkaline cellulose with monochloroacetic acid or its sodium salt in an alkaline medium [1].

CMC has received the greatest of all water-soluble cellulose ethers of practical importance, its production is organized in most industrialized countries [2]. Of the cellulose ethers produced on an industrial scale, the largest and most widely used is CMC, which has a degree of substitution of 0.6–0.9.

CMC is the most commonly used chemical, and it surrounds us almost everywhere. CMC is inexpensive to manufacture, non-toxic and flammable. Due to its ability to «work» in a wide temperature range, dissolve in aqueous

solutions and form a viscous jelly-like substance, it has been widely used in various industries [2]:

- in the oil and gas industry;
- in the construction industry;
- in the mining and processing industry;
- in the perfumery and cosmetic industry;
- in the textile industry;
- in the food industry and others.

The review [3] contains detailed information on the synthesis of CMC, provides dozens of technological directions for the use of CMC and some directions for the future use of CMC.

However, the use of CMC in these areas is highly dependent on purity, degree of polymerization, degree of substitution and homogeneity, which determine the characteristics of the resulting products, such as solubility, particle

size, viscosity, rheological properties, etc. Typically, CMC products are classified into three different grades: food grade, pharmaceutical grade, and industrial grade CMC based on purity and degree of substitution [4]. Currently, there are works devoted to the synthesis of CMC from waste of cellulose raw materials, as well as the use of materials based on agricultural waste, biomass due to their availability and low cost [5].

In the chemical field, many methods for the preparation of CMC are described, but they are all divided into two groups: solid-phase and suspension methods. The solid phase is obtained in industry without solvents, and in suspension in their presence.

The technological process for obtaining carboxymethyl cellulose consists of the following stages [1]:

1. Treatment of cellulose with alkali.
2. Processing with powder of monochloroacetic acid (MCA) or Na-MCA.
3. Carboxylation of cellulose.
4. Drying.
5. Grinding.

One of the main advantages of the solid-phase method for producing CMC is the simplicity of the hardware design, the absence of production waste, and fire safety.

The disadvantage of the solid-phase method is the complexity of predicting the parameters of the final product. The reason for this is the lack of sufficient possibilities to control the quality of the product in the process of carboxymethylation, associated with the complexity of controlling the temperature of the process, on which the parameter of the degree of polymerization depends [6, 7].

This is due to the large thermal effect, the high rate of the carboxymethylation process at elevated temperatures, and the complexity of heat removal. To solve this problem, data on the effect of temperature on the kinetics of the carboxymethylation process under industrial conditions are required [6].

An important indicator of the quality of sodium carboxymethyl cellulose, which determines the field of its application, is the degree of polymerization. However, obtaining a product with a specific parameter under industrial conditions is associated with a number of difficulties. Therefore, today the task is to improve the technology for the manufacture of sodium carboxymethyl cellulose in order to obtain a product with a given, as well as an increased degree of polymerization.

In [8], a new method for the synthesis of CMC was considered, in which cellulose was converted into an intermediate product of cellulose triacetate. The resulting product was converted to CMC using alkali.

However, the cited works do not consider the kinetics of CMC formation. During the synthesis of CMC, three chemical reactions occur, sequentially parallel. The synthesis process usually takes place in tubular reactors equipped with screw devices that serve to move the reaction mass [9].

Therefore, the development of a mathematical model of the kinetics of CMC synthesis, experimental studies to determine the rate constants of synthesis reactions, modeling of a screw reactor for CMC synthesis, and computer studies are relevant. *The object of the study* is the CMC synthesis reactor, and *the purpose of this work* is to develop a mathematical model of the synthesis reactor based on the model of the kinetics of CMC production.

## 2. Methods of research

The chemical essence of all technological processes for obtaining carboxymethyl cellulose consists in obtaining alkaline cellulose R-ONa (reaction (1)), in the interaction of alkaline cellulose with sodium monochloroacetate (SMCA) or monochloroacetic acid (reaction (2)) to obtain CMC. During the carboxymethylation of cellulose, the main reaction of the formation of CMC, NaCl, H<sub>2</sub>O and a side reaction of the hydrolysis of SMCA with the formation of sodium glycolate (HNa) and NaCl (reaction (3)) [1]:



Or in the form of a formal kinetic diagram:



The kinetics of the process is known to be the basis for modeling a chemical reactor. Therefore, the accuracy of all subsequent calculations depends on the accuracy with which the kinetic equations describe the process. The interaction of reagents in the preparation of CMC is a complex process that includes all stages of any heterogeneous process [10]. When studying the kinetics of CMC reactions, one of the possible approaches is to use a quasi-homogeneous model [10, 11], which is widely used in modeling processes on a catalyst grain. This approach can be used to describe and analyze individual stages; however, in the case of heterogeneous reactions of cellulose, a number of difficulties arise. In the course of these reactions, the properties of the solid phase and the processes change, respectively, they are non-stationary in time. The reaction does not take place on the surface of hard particles, but in the entire volume of the fibers.

If to assume that the concentration and reactivity of cellulose hydroxides, water, and products formed during the reaction remain approximately constant, then the use of a quasi-homogeneous model is quite acceptable and does not cause additional mathematical difficulties.

Based on the method of stationary concentrations [12], let's write down expressions for the rate of change in the degree of conversion arising from the mechanism of CMC formation (the key component of the reaction is NaOH).

From equation (4) let's obtain:

$$\begin{aligned} k_1 \{[\text{R-OH}] - [\text{R-ONa}]\} \{[\text{NaOH}] - [\text{R-ONa}]\} = \\ = k_2 [\text{R-ONa}] [\text{H}_2\text{O}], \end{aligned} \quad (7)$$

where  $k_1$  – direct reaction rate constant;  $k_2$  – response rate constant.

$$\begin{aligned} k_p = \frac{k_1}{k_2 [\text{H}_2\text{O}]} = \\ = \frac{[\text{R-ONa}]}{\{[\text{R-OH}] - [\text{R-ONa}]\} \{[\text{NaOH}] - [\text{R-ONa}]\}}, \end{aligned} \quad (8)$$

or, introducing new values:

$$k_p = \frac{x}{(y-x)(z-x)}, \quad (9)$$

where  $k_p$  – equilibrium constant of reaction (4);  $x$  – concentration of R-ONa, due to the degree of alkali conversion;  $y$  – concentration of cellulose;  $z$  – concentration of alkali;  $S$  – degree of MCA conversion in the main reaction.

Solving for  $x$ , let's obtain:

$$x^2 - x(y+z+k_0) + yz = 0, \quad (10)$$

where  $k_0 = 1/k_p$ .

Solving the resulting quadratic equation, let's obtain:

$$x = \frac{y+z+k_0}{2} - \sqrt{\left(\frac{y+z+k_0}{2}\right)^2 - yz}. \quad (11)$$

From equations (5) and (6), according to the law of mass action, let's find:

$$\frac{dC_{\text{CMC}}}{d\tau} = k_3 C_{\text{CMC}} \left( \frac{C_{\text{SMCA}}}{C_{\text{NaOH}}} - x - y \right) = k_3 C_{\text{CMC}} S, \quad (12)$$

$$\frac{dC_{\text{HNa}}}{d\tau} = k_4 \left( \frac{C_{\text{NaOH}}}{C_{\text{NaOH}}} - x \right) S = k_4 (z - y) S, \quad (13)$$

where

$$S = (C_{\text{SMCA}}/C_{\text{NaOH}} - x - y) = (b - x - y), \quad (14)$$

$$y = (C_{\text{R-OH}}/C_{\text{NaOH}} - x) = (a - x), \quad (15)$$

$$z = (C_{\text{NaOH}}/C_{\text{NaOH}} - x - y) = (1 - x - y). \quad (16)$$

To determine the values of the kinetic constants  $k_1$ ,  $k_2$  and  $k_0$  an experiment was carried out in an integral isothermal reactor. During the experiments, the degree of substitution of CMC and the concentration of free alkali were measured. The experiment was carried out in the temperature range 303–328 K. The apparatus, with a capacity of 0.5 l, was equipped with a conventional paddle stirrer. The preliminary mixing of alkaline cellulose with SMCA was carried out in a laboratory mixer. The synthesis time was 1.5–6 hours. In total, two parallel studies of 12 experiments were carried out. Four points were used in one study to determine each rate constant. As a result of these experiments, according to the obtained integral curves, the method of least squares was used to find the constants. To process the experimental data, let's use the linearized Arrhenius equation in a logarithmic form. The data obtained were used to plot straight lines in Arrhenius coordinates  $(\ln k) - (1/T)$ . To assess the adequacy of the obtained Fisher regression equation, the total variance  $S_y^2$  was compared with the final  $S_{\text{ost}}^2$ . As a result of the numerical implementation of the search problem, the following values of the constants and activation energies were obtained:

$$\ln k_0 = 4.90, \quad E_0 = 5.28 \text{ kcal/mol};$$

$$\ln k_3 = 45.50, \quad E_3 = 30.00 \text{ kcal/mol};$$

$$\ln k_4 = 48.60, \quad E_4 = 29.60 \text{ kcal/mol}.$$

After determining the reaction rate constants, one can proceed to mathematical modeling of the apparatus for obtaining CMC.

### 3. Research results and discussion

As a reactor for the synthesis of CMC, tubular reactors with a heat exchange jacket with a screw device inside are used. The reaction mixture with slight stirring moves to the outlet of the reactor using a screw device at a given velocity (residence time). There is practically no back mixing in the screw reactor. Therefore, according to the design of the reactor, let's assume that the hydrodynamic environment can be characterized as a model of ideal displacement along the length of the reactor [13].

It can be assumed that the mode of movement of the coolant flow in the jacket of the apparatus also corresponds to the ideal displacement mode. To calculate the residence time of the mixture in the reactor, let's apply the formula:

$$\tau_k = \frac{1}{v} \frac{\pi d^2 L}{4 \varphi}, \quad (17)$$

where  $v$  – volumetric velocity of the reaction mixture, m<sup>3</sup>/hour;  $d$  – diameter of the apparatus, m;  $L$  – length of the apparatus, m;  $\varphi$  – degree of filling the reactor with the reaction mixture.

The mathematical model of an isothermal CMC synthesis reactor will look like this [14, 15]:

$$\frac{dx}{d\tau} = k_3 x S \tau_k, \quad (18)$$

$$\frac{dy}{d\tau} = k_4 (z - y) S \tau_k. \quad (19)$$

The mathematical model of the polytropic CMC synthesis reactor will look like this:

$$\frac{dx}{dl} = \exp\left(\ln k_3 - \frac{E}{RT}\right) x S \tau_k, \quad (20)$$

$$\frac{dy}{dl} = \exp\left(\ln k_4 - \frac{E}{RT}\right) (z - y) S \tau_k, \quad (21)$$

$$\frac{dT}{dl} = \frac{\Delta H}{c_v} \left( \frac{dx}{dl} + \frac{dy}{dl} \right) - B_R (T - T_T), \quad (22)$$

$$\frac{dT_T}{dl} = B_T (T - T_T). \quad (23)$$

The initial conditions for the system of equations (15)–(20):

$$x(0) = x_0; \quad y(0) = y_0; \quad T(0) = T_0; \quad T_T(0) = T_{T0}.$$

In equation (22), the heat transfer parameter for the reaction volume:

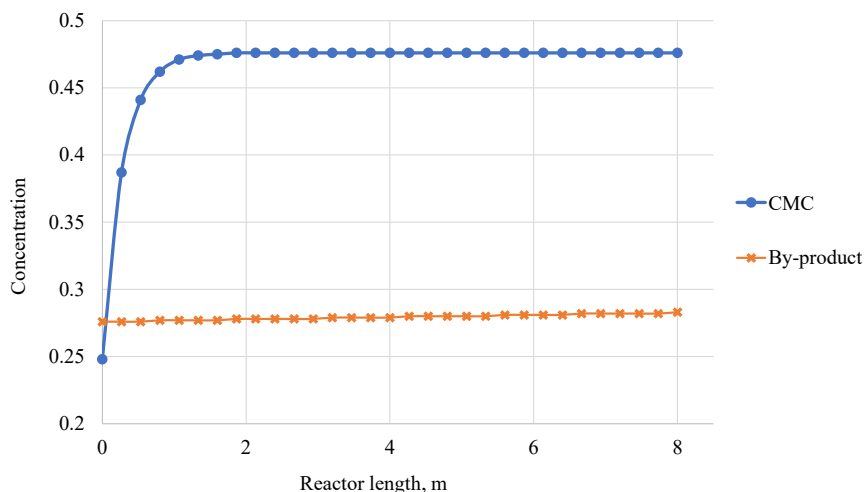
$$B_R = 4K_T T \tau_k / c_v d.$$

In equation (23), the heat transfer parameter for the coolant is:

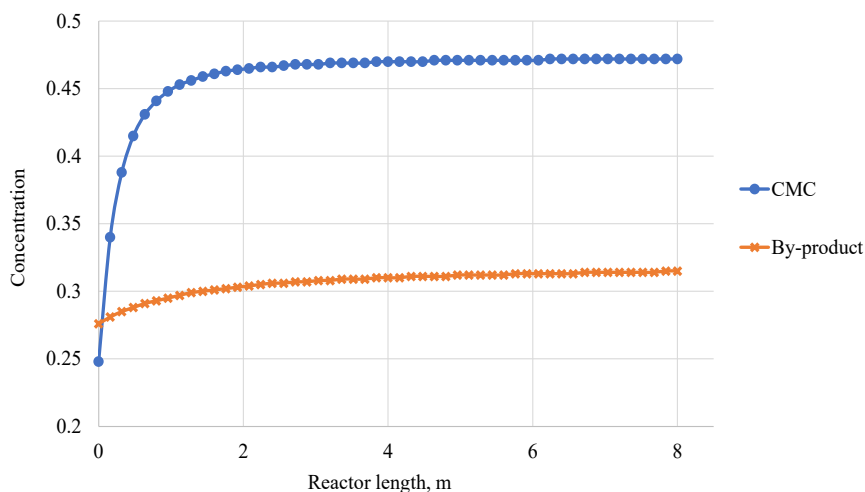
$$B_T = K_T \pi L d / c_T v_T.$$

Calculations using the isothermal reactor model (18), (19) and the polytropic reactor model (20)–(23) with the corresponding initial conditions were performed in the MathCAD package. Fig. 1 and Fig. 2 show the obtained concentration curves for CMC and sodium glycolate by-product. The curves obtained are identical, that is, the shown polytropic regime is close to isothermal. Fig. 3

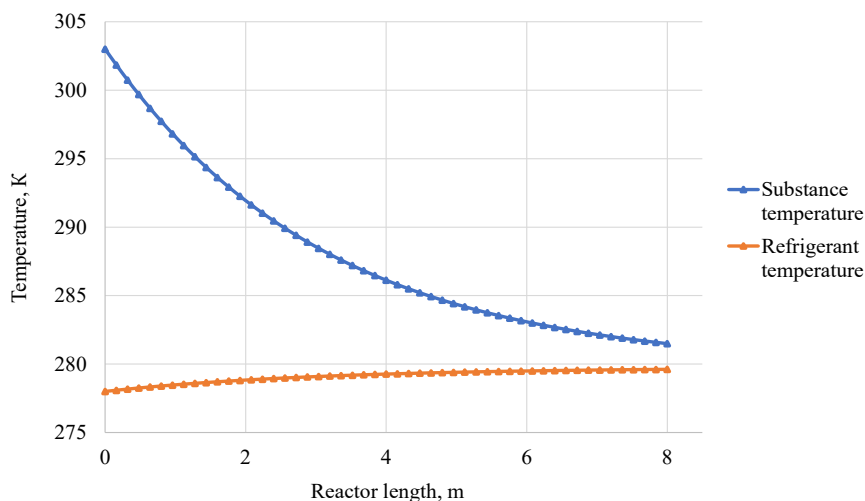
shows the change in the temperatures of the reaction mixture and the refrigerant. As it is possible to see, the reaction of CMC synthesis should be carried out in a mode close to isothermal, since the temperature of the reaction mixture at the initial moment of time can be very high. This can lead to carbonization of the cellulosic raw materials.



**Fig. 1.** Change in the concentration of the main and by-product along the length of the reactor in isothermal mode



**Fig. 2.** Changes in the concentrations of the main and by-product along the length of the reactor in the polytropic regime



**Fig. 3.** Change in temperature along the length of the reactor in the polytropic regime

The influence of the temperature of the inlet reaction mixture on the change in the concentration of CMC was also investigated. For this, the system of equations of the polytropic reactor (20)–(23) was calculated at different temperatures of the inlet reaction mixture in the range 283–303 K (Fig. 4).

The influence of the temperature of the refrigerant 272–287 K on the change in the concentration of CMC was investigated in a similar way (Fig. 5).

As seen from Fig. 5, at different inlet temperatures of the coolant, the process practically reaches a stationary mode already at a reactor length of about 3 m. And Fig. 4 shows that the CMC synthesis reaction proceeds along the entire length of the reactor, the size of which is 8 m.

The rate constant of the synthesis reaction depends on the process conditions. Using the proposed approach to describing the interaction of cellulose with a reagent, the reaction mixture considered as a quasi-homogeneous system can be described using a single-phase flow model.

The kinetic model of the process of CMC synthesis was developed on the basis of an experimental study of the process, taking into account changes in the conditions of the process on the basis of modern technology for the synthesis of CMC.

The results obtained on studying the kinetics of CMC synthesis and modeling a polytropic reactor adequately describe the process of CMC synthesis at a reaction mixture temperature of 283–328 K.

This approach to modeling kinetics can be used in the synthesis of other cellulose ethers: hydroxyethyl cellulose, cellulose acetate, methyl cellulose, which are currently widely used in various industries.

#### 4. Conclusions

The process of CMC synthesis in a screw tubular apparatus (apparatus dimensions: length 8 m, diameter 1.5 m) was investigated.

It was shown that during the synthesis of CMC, three successively parallel chemical reactions occur.

A mathematical model of the kinetics of CMC synthesis has been developed on the basis of a quasi-homogeneous model used in modeling processes on a catalyst grain.

On the basis of experimental studies, the rate constants of chemical reactions of CMC synthesis and the corresponding activation energies were obtained. The rate constants and activation energies for chemical reactions (5) and (6) are in good agreement with the constants given in the literature. The rate constant and activation energy for the reverse chemical reaction (4) were obtained for the first time.

The rate constant and activation energy for the reverse chemical reaction (4) were obtained for the first time.

A complete mathematical model of the polytropic process of CMC production has been developed, which includes four equations: two equations of material balance and two equations of heat balance.

The influence of the temperature of the reaction mixture and the temperature of the coolant on the yield of the main CMC product was estimated.

The influence of the temperature of the inlet mixture 283–303 K does not significantly affect the yield of the CMC reaction. The synthesis reaction with the required CMC concentration practically ends at a reactor length of 8 m. At a higher temperature, the destruction of cellulose can occur as an undesirable effect.

For the investigated range of variation of the variables, the refrigerant temperature has the greatest influence on the rate of CMC formation.

The calculations were performed in the MathCAD computer environment.

This model can be recommended for use in the design of industrial reactors of this type in the production of CMC by the solid-phase method and in the control of the operation of these reactors.

This kinetic modeling approach can be used in the synthesis of other cellulose ethers that are now widely used in various industries.

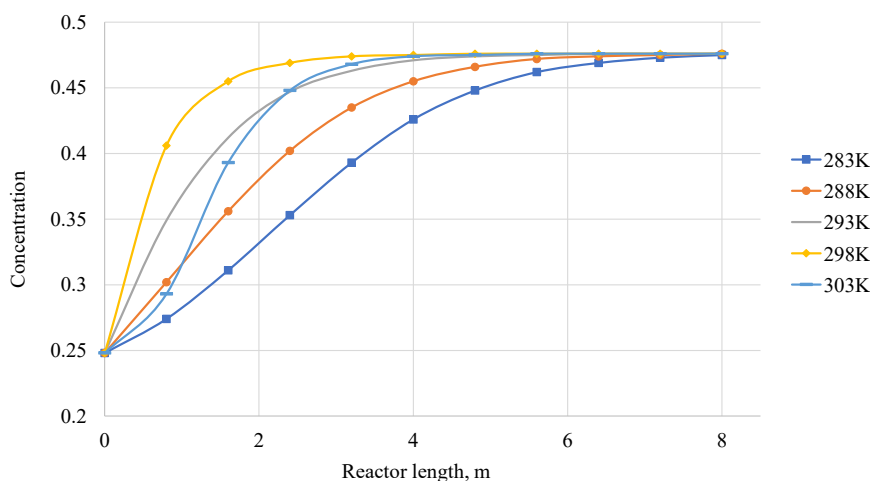


Fig. 4. Change in the concentration of CMC along the length of the reactor at different temperatures of the inlet mixture

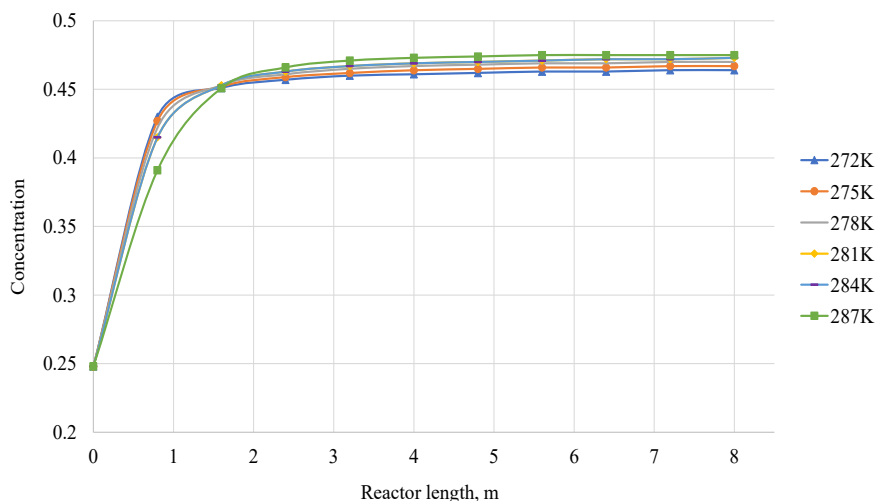


Fig. 5. Change in the concentration of CMC along the length of the reactor at different temperatures of the coolant



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