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## EVALUATION OF PHYSICO-CHEMICAL PROPRIETIES OF CARBOXYMETHYL CELLULOSE QUENCHING ENVIRONMENT DEPENDING ON THE DEGREE OF THERMAL DEGRADATION

Among the synthetic media used in the industry the most advantageous are the substances resulting as secondary products from the wood or paper processing industry, as carboxymethyl cellulose. Carboxymethyl cellulose dissolved in water is a synthetic quenching environment used in large tempering basin. Due to the dimensions of the quenching basins, the quenching medium is used repeatedly, which leads to its thermal degradation. The degree of modification of the cooling properties and corrosion behavior of aqueous solutions of carboxymethyl cellulose in the thermal range 800-50°C were studied. The changes in the physicochemical properties such as density, pH, specific heat, diffusivity were studied. These characteristics were found to exert major effect on cooling characteristics during tempering. At the same time, the technological characteristics of cooling were also studied. The specific cooling curve of the medium and thermally degraded compared with the characteristics of classic quenching media such as water and heat treatment oil was analyzed. This study provides guidance on maintaining the specific cooling characteristics of a tempering medium after repeated uses.

*Keyword:* Carboxymethyl cellulose; corrosion; heat transfer

### 1. Introduction

The synthetic quenching environment are substitutes for the classical ones such as heat treatment oil, water and salt baths, having certain advantages compared to them [1].

Thus, synthetic quenching environment are neither flammable nor highly polluting with the environment, like oil. They can be easily transformed by adding soluble or insoluble substances in order to improve the cooling properties in the requested thermal ranges, or their properties can be preserved for a longer time [2].

The additives added to the synthetic quenching environment have the role of disrupting and shortening the boiling regime with the formation of a vapor film. This effect is also accompanied by the attenuation of the thermal shock of the transition from the period of heating to the period of boiling with bubbles, shock, which is the cause of the majority of tempering defects [3,4].

Through evaporation, the salts are deposited on the surface of the parts and a greater number of vaporization centers are formed that accelerate the heat exchange. Among the synthetic

quenching environment used in the industry, the most advantageous are materials resulting as secondary products from the wood or paper manufacturing industry. Carboxymethyl cellulose is one of the substances that are considered residues in paper manufacture [5].

Carboxymethyl cellulose is part of the substances that represent waste in the manufacture of paper from organic raw materials such as reed. From an organic point of view, the substance is stable and is found in fibrous form. Dissolved in water in a percentage between 1.5 and 4.5%, it forms a good tempering medium [6]. Since it is an organic substance, the physicochemical characteristics degrade over time, leading either to a precipitation of the substance at the base of the cooling tank, or to biological degradation, or to physical degradation through repeated heating, TABLE 1 [7,8]. To overcome this, preservatives may be added or mechanical stirrers may be used to homogenize the substance in the tempering bath [9].

The work studied the variation of cooling properties in a non-agitated environment, as well as the corrosion characteristics of quenching media, namely 2.5% carboxymethyl cellulose solution dissolved in water.

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Characterisation of a quenching environment

Physical-chemical characteristics	Unit of measurement	Thermo-physical characteristics	Unit of measurement
Acidity	[pH]	Cooling curve T	
Iodine value	[g/100g oil]	Cooling rate variation	[°C/s]
Thermal conductivity $\lambda$	[w/mK]	Heat transfer coefficient per thermal interval $\alpha$	[w/m <sup>2</sup> K]
Surface tension – wetting angle	[°]	Cooling intensity $H$	[m <sup>-1</sup> ]
Viscosity – $\eta$ – dynamic – $\gamma$ – kinematic	[cP] [cSt]	Boiling temperature	[°C]
Density $\varphi$	[kg/m <sup>3</sup> ]	Ignition temperature (flammability $T$ )	[°C]
Thermal diffusivity	[m <sup>2</sup> /s]	Thermal stability	
Specific heat C	[J/kg·K]		

TABLE 2

Chemical composition of steel, [%]

Elements	C	Si	Mn	P	S	Cr	Ni	Cu	Fe
Percentage	0.389	0.26	0.672	0.0072	0.0125	0.055	0.011	0.053	rest

## 2. Materials and methods

CMC is a vegetable component resulting as a by-product from paper production. It is a partially soluble substance in water with a fibrous structure having inverse solubility in water, that is, up to 300°C it is in polymeric form and below this temperature it dissolves in water. The chemical formula is C<sub>6</sub>H<sub>16</sub>O<sub>8</sub> [10].

The material used for linear voltammetry, is the carbon steel with the chemical composition TABLE 2.

The effect of thermal fatigue on the thermo-physical characteristics and the cooling capacity of the synthetic quenching medium type carboxymethyl cellulose solution in water was analyzed. Fatigue tests were done for an amount of heat transferred from the Kantal resistor and the number of cooling cycles 35. Amount of transferred heat,  $Q$  for 35 heating cooling cycles is 1274000 [J] (text in German language).

The results obtained must be related to the amount of heat transferred, because the thermal fatigue test of liquids is not standardized and we cannot otherwise compare the results (a universal size must be taken as a term of comparison).

Characterization of the cooling curves, physical characteristics, density, remaining non-evaporated mass, specific heat, thermal diffusivity. In the paper, the cooling curves of a standardized cylindrical silver sample were drawn. The experiment was carried out using an installation made for this purpose. The specimen was heated to a temperature higher than 800°C and inserted into the container with the analyzed quenching medium, the cooling curve was recorded with a “y-t recorder”.

For each cooling medium were calculated:

- speed of cooling on intervals [°C/s];
- the thermal transfer coefficient on intervals.

$$\alpha_i = \frac{3600 \cdot m \cdot c}{\Delta t_i \cdot S} \ln \frac{T_i - T_o}{T_f - T_o} \left[ \text{w/m}^2\text{k} \right]$$

$$\alpha_g = \frac{\alpha_1 \cdot \Delta t_1 + \dots + \alpha_g \cdot \Delta t_g}{t_{total}}$$

where:

$m = 0,0399$  [Kg] mass of the specimen,

$c = 0.056$  [Kcal / Kg·grad] the specific heat of Ag,

$S = 0.001408$  [m<sup>2</sup>] area of the specimen,

$\Delta t$  [s] Time interval,

$T_i, T_f$  [°C] initial and final temperature per interval,

$T_0$  the initial temperature of the cooling medium;

- cooling intensity

$$H = \frac{\alpha_g}{2\lambda} \left[ \text{m}^{-1} \right]$$

$$\lambda_{Ag} = 418.5 \text{ W/mK}$$

Corrosion behavior was (analyzed) by rapid electrochemical tests and dynamic potentiometry. Open circuit potential and dynamic polarization measurements were performed using a Volta Lab 21 potentiostat (Radiometer, Copenhagen). The procedure was controlled by VoltaMaster4 electrochemical software. After corrosion analysis in fresh and altered environment, scanning electron microscopy (SEM, VegaTescan LMH II, SE detector, 30 kV) has been performed. The surface chemical composition has been established with an EDS detector, Bruker X-flash.

Contact angle measurements were carried out using a Kruss Easy Drop goniometer. The sessile drop method was selected to measure the static contact angles of distilled water and CMC solutions. Using Drop Shape Analysis software (DSA version 1.90.0.14) the average of the right and left contact angles was then calculated.

## 3. Results and Discussion

### 3.1. CMC microstructural and chemical characterization

Carboxymethyl cellulose is a substance of plant origin obtained as a by-product in the processing of paper from plant

tissue. The substance is partially miscible in water, forming solutions with multiple industrial uses, such as a cooling medium for tempering at medium speeds [11]. Being a substance of biological origin, it has the property of changing its physico-chemical characteristics after the heating-cooling cycle, degrading.

The Fig. 1a) and 1b) show the structure of the undissolved CMC, the structure being fibrous in nature. From a chemical point of view, it contains C, O and H and elements such as Na and Cl. The EDX image shows the distribution of the element in the fibrous structure [12].

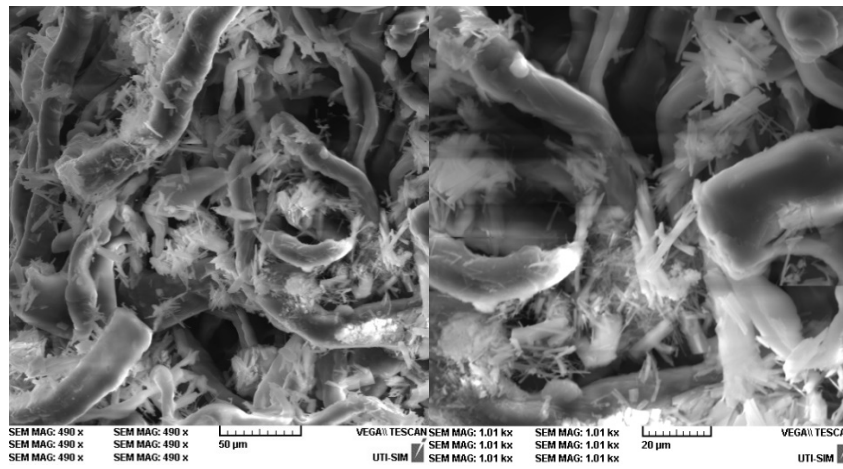
CMC of low DS (0.2-0.7) has free hydroxyl groups, which can be further esterified by organic acid anhydrides to attain either single or mixed ester derivatives. The sodium salts of such

carboxyalkyl acetyl celluloses are dissolved uniformly in water and in aqueous organic solvents.

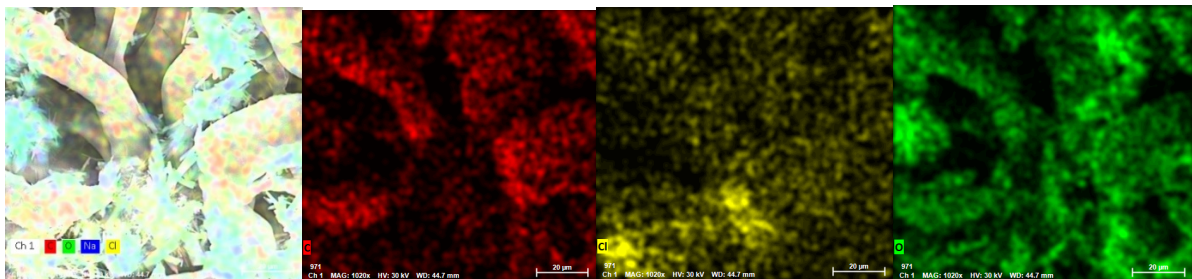
### 3.2. Thermal properties

On the basis of the obtained results the following curves were drawn:

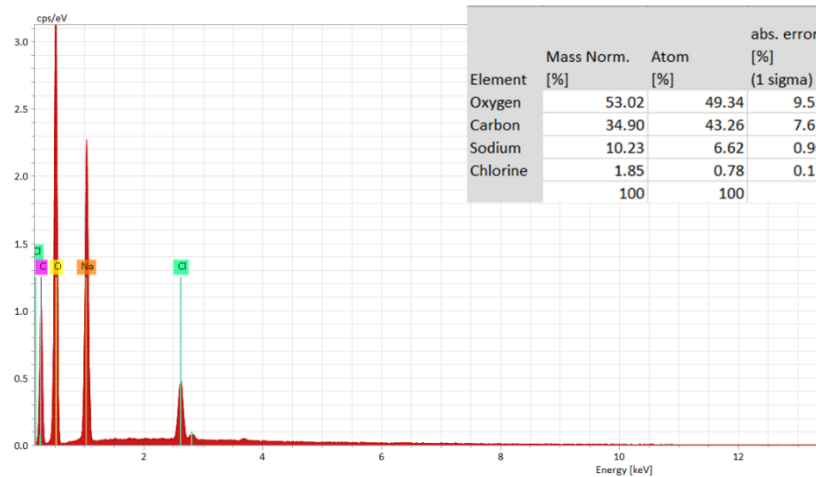
- Cooling curves  $T = f(t)$ , Fig. 2a);
- Variation of cooling rate with temperature  $v_r = f(T)$ , Fig. 2b);
- Variation of the heat transfer coefficient with temperature  $\alpha_i = f(T)$ ;
- Cooling intensity  $H = \alpha_g / 2\lambda$ , Fig. 3.



a)



b)



c)

Fig. 1. a) Scanning Electron Microscopy (SEM) micrographs, fibrous structure of CMC, b) EDAX analysis of CMC fiber, c) EDS spectra

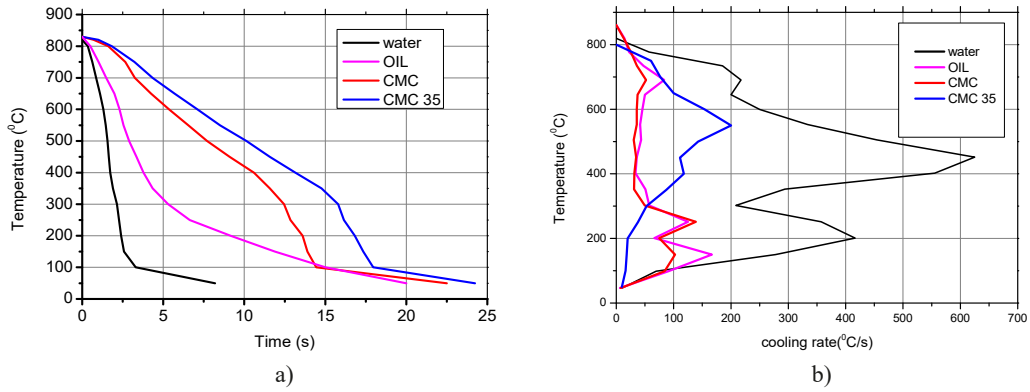


Fig. 2. a) Cooling curves of CMC and CMC 35, b) Cooling rates on ranges of classical media and CMC and CMC 35

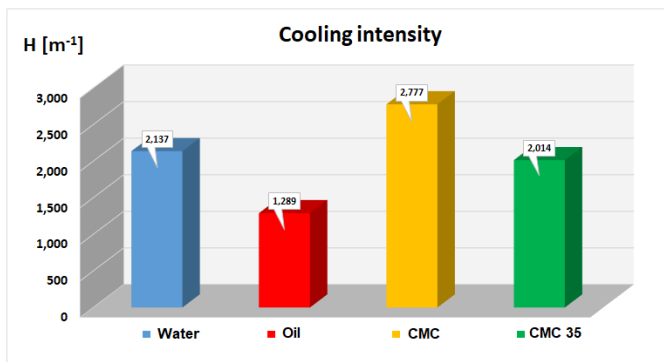


Fig. 3. Cooling intensity over the thermal range 800-50C of water, thermal oil, CMC and CMC35

TABLE 3

Cooling parameters

Sample	$\alpha_g, w/m^2K$	$H, m^{-1}$
Water	1788.937	2.137
Oil	1079.445	1.289
CMC	2324.452	2.777
CMC 35	1685.79306	2.01409

### 3.3. The amount of mass evaporated

After performing each set of experiments, the amount of liquid remaining in the container was measured with an analyti-

cal balance and by difference the amount of liquid evaporated, knowing that initially the mass of the liquid was 1000 g, Fig. 4.

The containers in Fig. 4 show the color of the liquids used in the experiment, namely the control liquid, carboxymethyl cellulose solution in unused water, and the carboxymethyl cellulose solution type liquid used thermally with 35 heating and cooling cycles. The darker color of the medium used is observed due to the presence of iron oxides and other contaminated substances.

After performing the 35 test cycles, the amount of liquid listed in the table remained in the container. The graph in Fig. 4 shows the variation of the amount of coolant after quenching and gives indications regarding the evaporation rate. The amount of liquid evaporated, i.e. the intensity of evaporation depends on the interval between the two coolings so that when cooling with a longer pause between them the liquid has time to cool down enough so that it can take the same amount of heat in a shorter time preventing excessive evaporation.

### 3.4. Measurement of density variation

The used coolant samples were also subjected to density measurements at 25°C using densimeters with the ranges of values 1.07÷1.14 g/cm<sup>3</sup> și 1÷1.07 g/cm<sup>3</sup>, Fig. 5.

The device was checked by measuring the density of distilled water at a temperature of 25°C ( $\rho_{water} = 0.9997 \text{ g/cm}^3$ ). The values of the measured densities were listed in the TABLE 4.

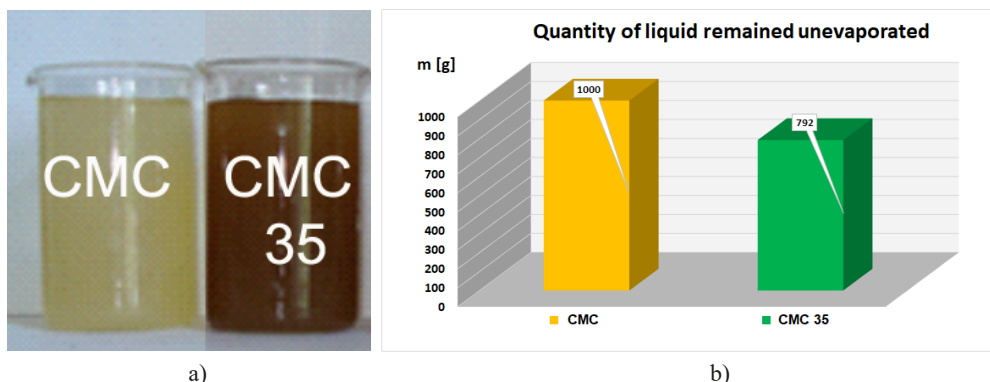


Fig. 4. a) Colour of CMC and CMC 35 heating media, b) Amount of liquid remaining unevaporated for CMC and CMC 35

TABLE 4

Density of used liquids on repeated cooling

No Cycles	Control Sample	35
Density, $\rho$ [g/cm <sup>3</sup> ]	1.007	1.009

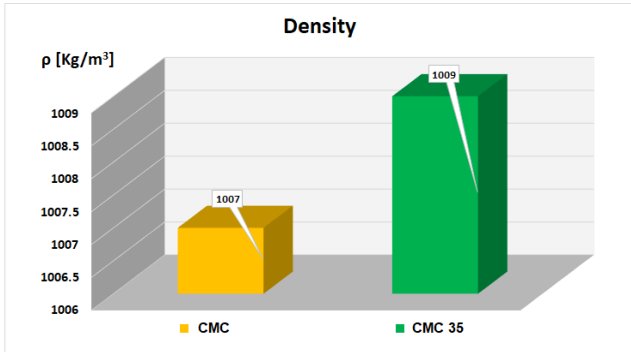


Fig. 5. Comparative density analysis of spent liquids at repeated cooling

It can be seen from the table, but more suggestively from the graph, that the density varies slightly unevenly and not strictly increasing as might have been assumed due to the continuous evaporation of the cooling medium, which is due to the amount of iron oxide (in the form of suspension) and uneven combustion of carboxymethyl cellulose.

#### 4. Thermal conductivity measurement

Thermal conductivity is a physical measure that describes the capacity of a material to conduct heat when subjected to a temperature difference. The thermal conductivity is dependent on the physical properties of the material such as the state of aggregation, the type of organic or inorganic material, density, compactness, porosity or fraction of voids, humidity, temperature.

The thermal conductivities of the used liquids were measured with a special device, listed in TABLE 5 and represented graphically in Fig. 6. The thermal conductivity determination equipment used is Mathis TCi, consisting of unit (model TCi-2-A, serial TH89-05-00092) and sensor (model/serial TCi92), (Fig. 1), C-Therm Technologies Ltd., Canada.

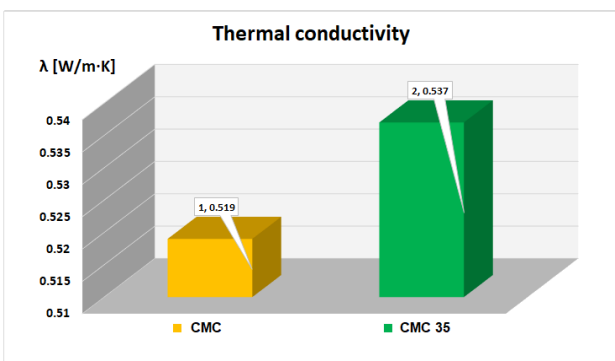


Fig. 6. Comparative analysis of thermal conductivity of spent liquids at repeated cooling

The TCi system provides data on thermal conductivity and thermal effusivity for different types of materials in different states of aggregation (solids, liquids, powders, pastes).

It is observed that the thermal conductivity varies slightly, increasing when the quenching medium with suspensions and solution is thermally used.

#### 4.1. Determination of specific heat

The determinations were made with the help of an installation with an automatic heating system and two brass enclosures with similar thermophysical characteristics. The calorimeter enclosure, was thermostated at 30°C, and distilled water was used as the comparison liquid. The two brass tubs inside which the liquid to be measured and the distilled water are placed are provided with a stirring system and heating elements.

The two brass vessels have the same weighed mass  $m_{v1} = m_{v2} = m_v = 323.8$  g and for the analyzes the same amount of working and reference liquid (measured with the analytical balance) was placed in each.  $m_l = m_{water} = 487$  g –  $323.8$  g =  $163.2$  g

The heat balance in the calorimeter is

$$R \cdot I \cdot \tau = m_l \cdot c_l (T_1^* - T_1) + m_v \cdot c_v (T_1^* - T_1) + Q_p \quad (1)$$

$$R \cdot I \cdot \tau = m_{water} \cdot c_{water} (T_2^* - T_2) + m_v \cdot c_v (T_2^* - T_2) + Q_p \quad (2)$$

where:

$R$  – electric heating resistance;

$I$  – current intensity;

$\tau$  – measurement time interval;

$m_v$  – the mass of the brass vessel ( $m_v = 323.8$  g);

$m_l$  – mass of the working liquid;  $m_a$  – mass of distilled water;  $m_l = m_a = m$ ;

$c_l$  – specific heat of the working fluid;

$c_a$  – specific heat of distilled water ( $c_a = 4185$  J/KgK);

$c_v$  – specific heat of brass ( $c_v = 380$  J/KgK);

$T_1$  – the initial and final temperatures of the measurement interval  $\tau$  for the working liquid;

$T_2$  – the initial and final temperatures of the measurement interval  $\tau$  for the reference liquid (distilled water);

$Q_p$  – the amount of heat of the enclosure.

From relations (1) and (2)

$$\begin{aligned} m_l \cdot c_l (T_1^* - T_1) + m_v \cdot c_v (T_1^* - T_1) &= \\ m_{water} \cdot c_{water} (T_2^* - T_2) + m_v \cdot c_v (T_2^* - T_2) &\Rightarrow \\ \Rightarrow c_l &= \left( \frac{T_2^* - T_2}{T_1^* - T_1} - 1 \right) \cdot \frac{m_v}{m} \cdot c_v + \frac{T_2^* - T_2}{T_1^* - T_1} \cdot c_{water} \end{aligned} \quad (3)$$

Based on the recorded data and with relation 3, the specific heats were calculated. The results of the calculations were drawn in Fig. 7.

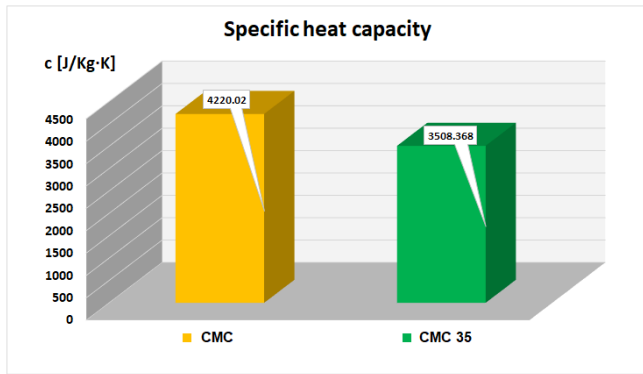


Fig. 7. Comparative analysis of specific heat for waste liquids at cooling

According to the measurements, the specific heat of the unused cooling liquid is maximum and decreases progressively the more the liquid is used, respectively at 35 cooling cycles.

#### 4.2. Thermal diffusivity

With the help of specific heat, density and thermal conductivity we can calculate the thermal diffusivity according to the formula:

$$a = \frac{\lambda}{\rho \cdot c_p} \text{ [m}^2\text{/s]} \quad (4)$$

where:

- $a$  – thermal diffusivity;
- $\rho$  – density;
- $\lambda$  – thermal conductivity;
- $c_p$  – specific heat.

All data were presented centrally in TABLE 5.

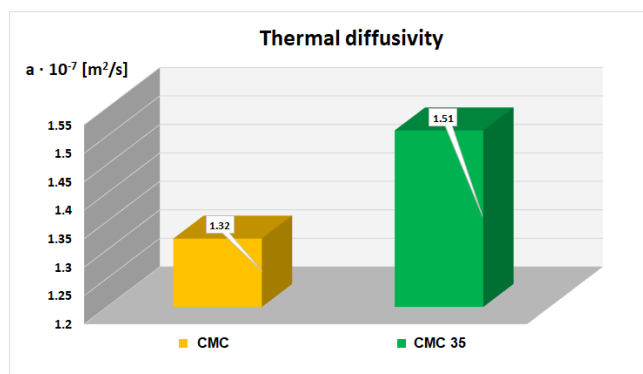


Fig. 8. Comparative analysis of thermal diffusivity for used liquids at cooling

#### 4.3. Contact angles

It can be seen from the analysis of the contact angles that with the thermal wear, a quenching medium with a more hydrophilic character is obtained – Fig. 9a and 9b, which favors the maintenance of a relatively stationary liquid film around the part, which slightly slows down the cooling [13].

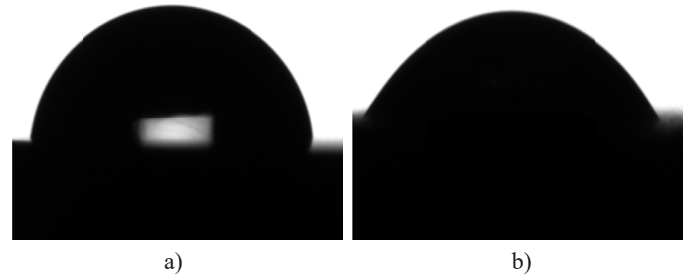


Fig. 9. Shape of the drops showing the contact angle at a) CMC and b) CMC 35

Contact angle of the CMC solution droplet, the angle of the droplet with the metal sample surface  $\theta = 75^\circ$  for unused CMC.

Contact angle of the CMC solution droplet with the sample surface  $\theta = 55^\circ$ , thermally fatigued CMC 35 heating-cooling cycles.

Along with the thermal wear of the cooling medium, a phenomenon of decomposition of the carboxymethylcellulose solution occurs, which changes the adhesion to the surface of the sample, increasing the hydrophilic character of the liquid substance.

This has the effect of causing the polymer solution to adhere to the surface of the sample, resulting in increased heating time, which slows cooling over large temperature ranges, which is unfavourable for the quality of the quenching environment.

#### 4.4. Electrochemical corrosion

For the evaluation of the electrochemical parameters of the corrosion process generated by the 2.5% CMC untreated and thermally fatigued quenching environment, Tafel diagrams [14,15] are presented in Fig. 10. In the case of linear voltammetry, the parameters according to which the degree of corrosion of a medium towards a specific metal can be evaluated are: the corrosion potential ( $E_{cor}$  [mV]); the anodic branch  $b_a$  [mV], which indicates the oxidizing character of the medium and the cathodic branch  $b_c$  [mV], which shows the reducing nature; the corrosion rate ( $V_{cor}$  [mm/year]) and current density ( $J_{cor}$  [mA/cm<sup>2</sup>]). From the Tafel diagram it can be seen that the anodic branch of the polarisation curve has a sharper slope than the cathodic branch in both cases.

TABLE 5

Thermal properties

Sample	$\lambda$ [W/mK]	$\rho$ [kg/m <sup>3</sup> ]	$c$ [J/kgK]	$a$ [m <sup>2</sup> /s]	Amount of liquid remaining unevaporated, $m$ [g]
Control sample	0.519	1.007	4220.02	$1.32 \times 10^{-7}$	1000
CMC 35	0.53775	1.009	3508.6385	$1.51 \times 10^{-7}$	792

TABLE 6

Corrosion parameters

Sample	$E_0$ , [mV]	$b_a$ , [mV]	$b_c$ , [mV]	$R_p$ , [ohm·cm <sup>2</sup> ]	$J_{cor}$ , [mA/cm <sup>2</sup> ]	$V_{cor}$ , [mm/Y]
CMC	-1025.1	754.3	167.3	770.57	0.0932	1.073
CMC35	1071.8	674.0	145.6	490.44	0.1065	1.226

The higher the current density ( $J_{cor}$ ), the stronger the intensity of the redox corrosion reaction and the more the metal surface will be affected by corrosion. The difference between the  $J_{cor}$  values of CMC and CMC 35 media can be explained by a higher corrosion tendency of CMC 35 media due to new compounds arising from thermal degradation.

The anodic oxidation phenomenon is much more intense in CMC 35 so thermally tired medium with 35 heating-cooling cycles compared to fresh CMC medium. This is highlighted by the value of the  $b_a$  anode slope which in thermally altered medium is 1023 mV and in clean medium is 773 mV.

The cathodic reduction phenomenon indicates a period of resistance to the corrosive agent by continuous replenishment of the existing protective boundary layer on the surface of the part to be corroded and which is indicated by the value of the cathodic slope  $b_c$  which is -399 mV in fresh medium and -380 mV in thermally altered environment.

With the breakdown of the protective layer indicated by the corrosion potential the corrosive metal liquid interface becomes thermochemically unstable and corrosion occurs.

The high value of the  $b_a$  constant, especially at thermally weathered CMC  $b_a$ , indicates that the anodic oxidation process is high, so the spent quenching environment has a strong oxidative effect.

The polarization resistance gives indications of the thermodynamic stability characteristics of the metal and

it is observed from TABLE 6 that metal-CMC interaction,  $R_p = 770.57$  ohm/cm<sup>2</sup>, while for CMC-worn-metal action,  $R_p = 490.44$  ohm/cm<sup>2</sup>, which highlights the corrosive nature of the thermally fatigued biologically based quenching medium.

From the structural analysis Fig. 11 (in figure is the contact angle) for the CMC-corroded steel enhancement sample, much less pitting is observed than for the CMC 35-corroded sample, Fig. 12. Also, it can be seen from Fig. 12a (Fig. 9 contains only a and b), conglomerate type deposits which are distributed both on the surface and inside the pitting. These are due to the presence of suspended particles assigned as residues from previous heating cycles. From Fig. 12a) and 12b), EDAX analysis and SEM micrographs, chlorine compounds are observed on the edges of the pitting holes due to CMC35, and intermetallic oxides are seen inside the holes.

TABLE 7

Chemical composition of CMC 35

Element	[wt.%]	[Norm. wt.%]	[Norm. at.%]	Error in %
Iron	94.19	88.27	67.85	2.41
Oxygen	9.92	9.29	24.95	1.51
Carbon	1.91	1.79	6.41	0.59
Chlorine	0.67	0.63	0.76	0.05
		100	100	

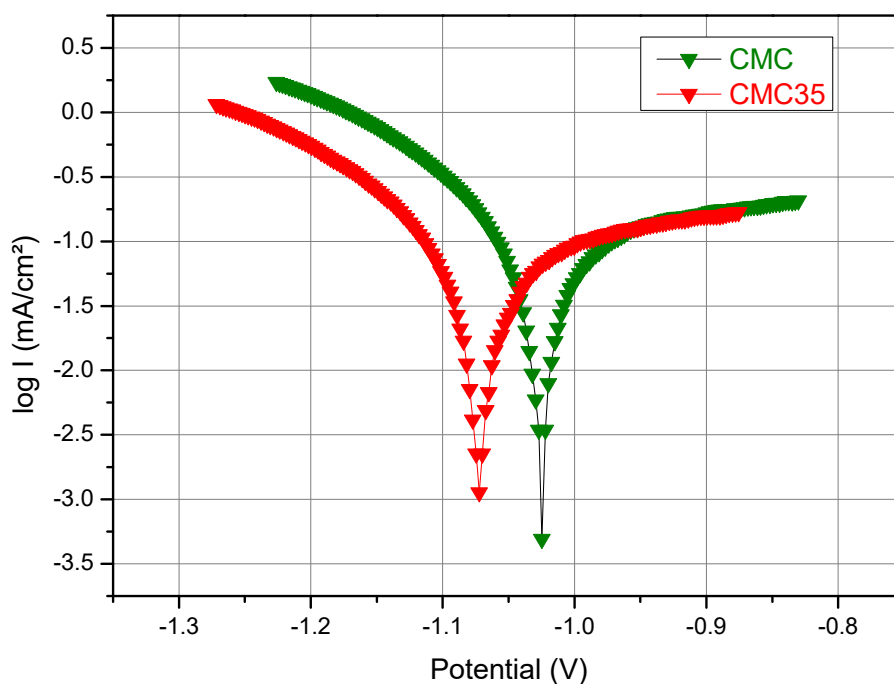
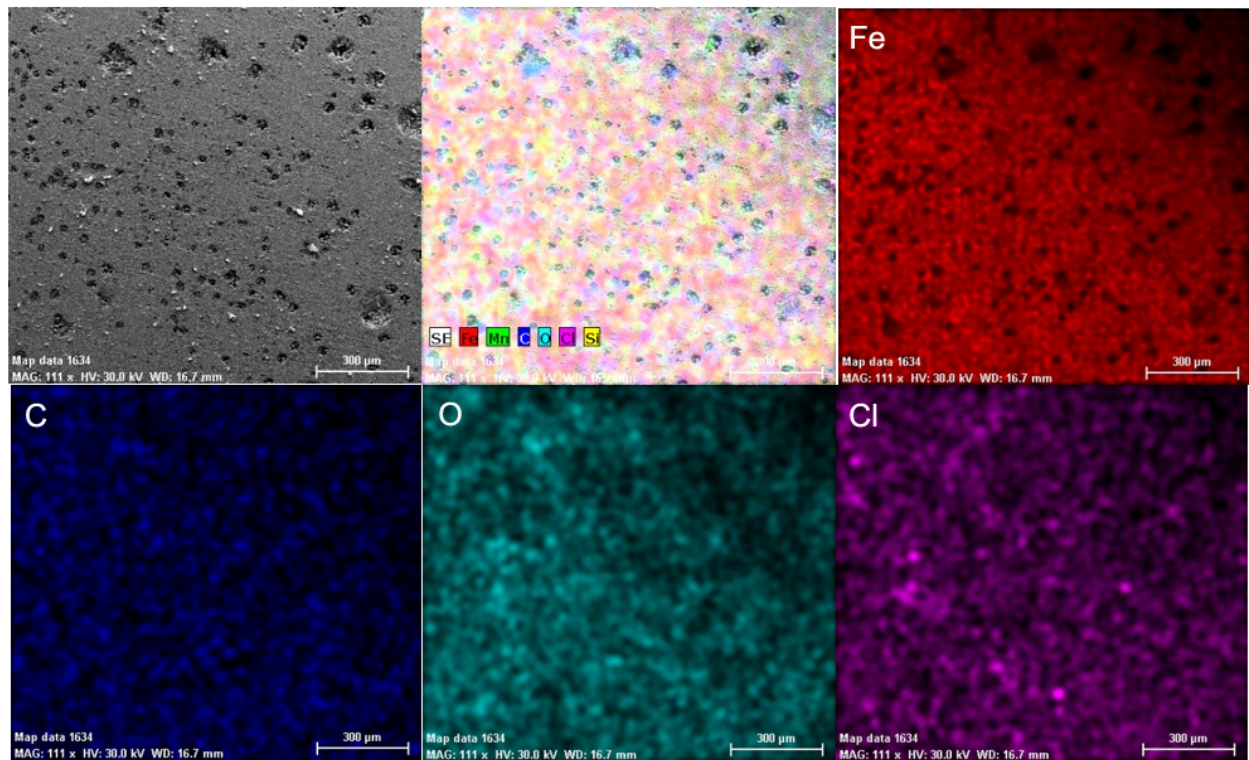
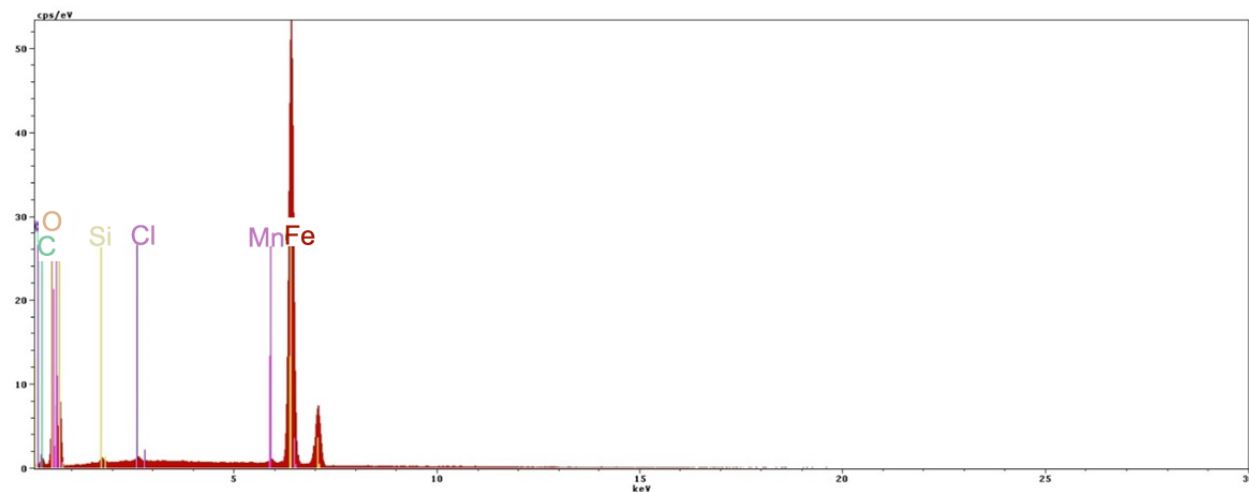


Fig. 10. Tafel diagrams obtained averaging CMC and CMC 35



a)



b)

Fig. 11. a) SEM and EDX analysis of CMC corroded steel sample, b) EDX spectrum

## 5. Conclusions

The solution of carboxymethyl cellulose in water has thermal degradation points around the temperature of 300°C, which means that after a number of heating-cooling cycles between 800 and 30°C it changes its physicochemical and technological heat absorption properties. Analysing the cooling characteristics of the fresh and thermally used CMC environment for quenching, it can be seen that the change in the characteristics of the environment following the heating-cooling cycles leads to a change in the cooling curve, resulting in a slower cooling environment.

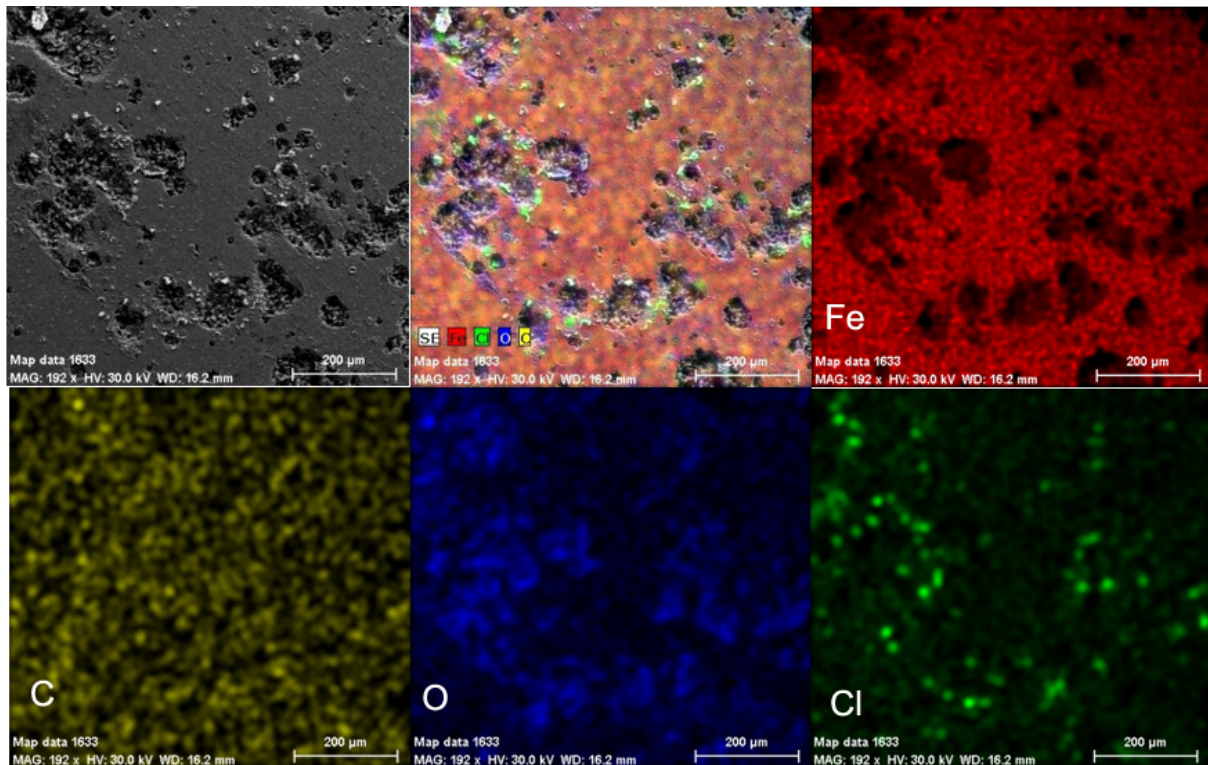
The CMC solution in water with thermal ageing becomes more hydrophilic than the initial solution leading to an increase

of the heating time and thus slowing down the cooling in the thermal range 800–400°C.

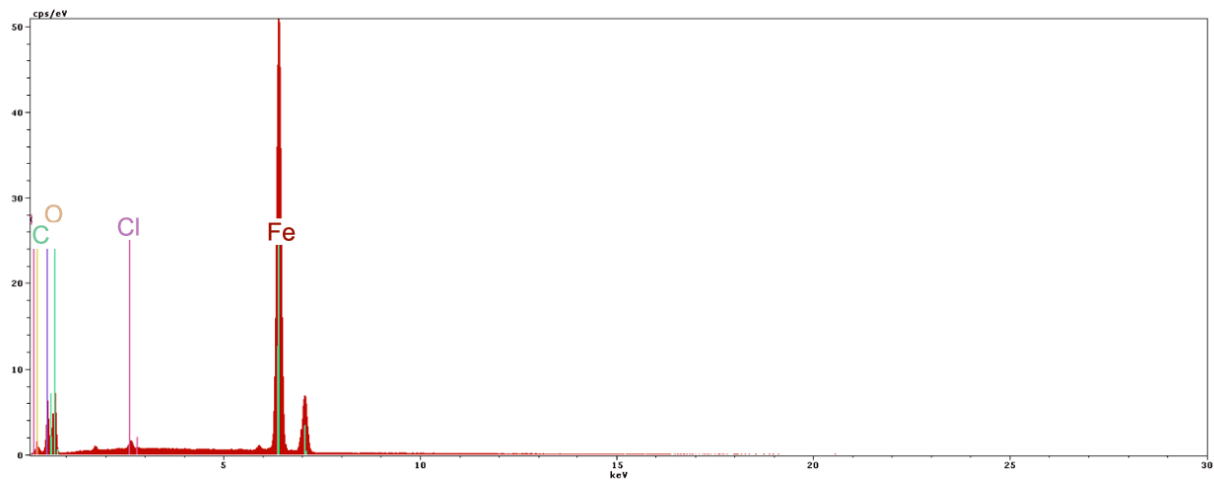
From the point of view of the corrosion characteristics, studying the Tafel curves we observe that a thermally weathered medium is more chemically aggressive, more easily tearing the protective boundary layer of an improving sample steel.

In cyclic corrosion, CMC35 has a much higher tendency than CMC to form corrosion pits, which is also evident in SEM and EDX analyses. The presence of iron oxides in the water has an important effect, due to cavitation cleaning and bubble boiling of the oxides on the surface of the samples subjected to repeated heating and cooling.





a) Add explanation of the elements



b)

Fig. 12. a) SEM and EDX analysis of the CMC 35 corroded upgrading steel sample, b) EDX spectrum

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