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ARTICLE

The Effect of Methacrylic Acid and Maleic Acid on the Isothermal Kinetics of Acrylic Acid Crosslinking Co-polymerization under Conventional and Microwave heating

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ABSTRACT

The kinetics of free-radical crosslinking co-polymerization (FRCCP) of acrylic acid (AA) with both methacrylic acid (MA) (PAMA hydrogel) and maleic acid (MAL) (PAMAL hydrogel) was investigated under the conditions of isothermal conventional heating (CH) and under the conditions of microwave heating (MWH) with controlled cooling. The kinetics curves of FRCCP of PAMA and PAMAL hydrogels under the conditions of CH are described with the kinetics model of second order chemical reaction, whereas the kinetics curves under the conditions of CH are described with the kinetics model of Polany-Winger. It is proved that MWH leads to the changes in the rate of FRCCP and to the changes in the values of the kinetic parameters (activation energy (Ea) and pre-exponential factor (lnA). It was found the existence of relationship between the values of the kinetic parameters calculated for MWH and CH for PAMA and PAMAL hydrogel synthesis process, which is well-known as compensation effect. The effect of MWH on the kinetics of FRCCP for PAMA and PAMAL hydrogel formation were explained by applying the model of activation by selective energy transfer (SET). The changes in kinetics model, rate of FRCCP and kinetics parameters, caused with the MWH can found wide application in designing novel technologies for obtaining polymers and for synthesis of polymers with novel physico-chemical properties. The suggested mechanism of activation for polymerisation under the conditions of MWH also enables development of novel reaction systems and technologies for polymers productions.

1. Introduction

Hydrogels present a novel class of non-metallic materials which attract significant attention during last two decades. Hydrogels are usually defined as 3-D networks of hydrophilic polymers which absorb huge amounts of water, water solutions and biological liquids without to dissolve neither to lose physical or chemical stability. Thanks to their distinguishing properties (hydrophilicity, biocompatibility, high swelling degree in water and water solutions, low or complete nontoxicity) and to capabilities to respond to different changes in medium by which are surrounded, hydrogels found widespread applications in different fields such as: bio-medical, pharmaceutical, agrochem-

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ical and environmental [1]. The most important method for hydrogel synthesis is free-radical crosslinking polymerizations (FRCP) under the conventional heating (CH) where the kinetics of hydrogel formation is followed isothermally [2].

Microwave heating (MWH) is nowadays well-known non-conventional source of energy which is applied in various field of chemistry, especially organic [3] and polymer synthesis [4], material science [5] and various physicochemical processes [6,7,8]. The advantages of using MWH in synthetic purposes are connected with the facts that: MWH considerably accelerates the reactions rate, enhances the yields of desired product giving products with better properties. Accordingly, application of microwaves appeals more attention [9]. The influence of microwaves on the kinetics of chemical reactions was predominantly explained by thermal effects (overheating, hot-spots, selective heating) as well as by specific microwave effects [9,10].

Among the earliest application of microwave heating was reported in 1968 [11] as unconventional method for poly(methyl methacrylate) (PMMA) processing as alternative to common processing using water-bath heating [12]. The initially used MWH to polymerize methyl methacrylate (MMA) exhibited substantial decrease in polymerization time from 9 hours to just 3 minutes, at the same time giving same properties of synthesized PMMA as in the case of CH [11,13,14]. A comprehensive review was nicely presented the actual literature overview and the perceptions of MWH in synthesis of polymers [15]. The atom transfer radical polymerizations (ATRP) of MMA under controlled MWH was performed and gave vary similar results to the ones obtained under CH which was a good indication that there is not “microwave effect” in the MWH ATRP. The considerable reactions rate acceleration over CH was explained [16]. A comparative analyses of the CH and MWH kinetics of MMA polymerization was performed, which reveals that the polymerization rates increased under the MWH up to 8.9 times. The kinetics parameters for MMA polymerization for the MWH conditions were 1.2-12 times lower than comparable for CH conditions and they were dependent on the degree of polymerization. The decreased value of activation energy (Ea) of MWH polymerization is explained with the non-equilibrium energetic distribution of the reactants [17]. Spasojevic and co-workers investigated the effects of isothermal heating modes, CH and MWH, on the kinetics of polymerization and the properties that are significant for the application of several PMMA base dental materials commercially available. It was found that the use of MWH in provokes the changes in the kinetics model of polymerization, the values of rates constants which are ~4 times higher than in the case of CH polymerization and the values of kinetics parameters (activation energy and pre-exponential factor) which are lower for the MWH polymerization [18,19].

In the literature are available reports about the preparation of superabsorbing materials under MWH from sodium acrylate, corn starch, and poly(ethylene glycol) diacrylate [20]. Superabsorbents were prepared by using sodium acrylate, starch, and 2-acrylamido-2-methylpropanosulfonic acid. Then, the copolymerization was more rapidly under MWH and the obtained products exhibited higher swelling rates than in the case of CH synthesized products [21]. The microwave-assisted preparation of poly (N-iso-propylacrylamide) (PNIPAAm) hydrogel with improved responsive properties was described. It was found that the MWH hydrogel synthesis under 75 W was finished in only 40 sec giving 99 % yield [22]. Z. Zhao and co-workers showed that the use of MWH considerably enhanced the reaction rate for PNIPAAm hydrogel synthesis from the hours to a few minutes and achieved the yields up to 99%. Additionally, the obtained PNIPAAm hydrogels synthesized under MWH had more porous structure and had higher swelling degree [23].

The grafting of acrylic acid (AA) onto chitosan under MWH was investigated. It was shown that the MWH leads to the increase in the reaction rate up to the 8 times in comparison to the CH method giving the grafting degree and grafting efficiency of the obtained product similarly to the grafting in CH method [24]. MWH has been successfully used to obtain chitosan-g-poly (acrylonitrile) without use of initiator during the only 1.5 min [25]. Graft copolymer of κ-caragenan with methyl methacrylate was synthesized with high yield by using potassium-persulfate as an initiator. It was confirmed that controlled MWH can be used for the preparation of stable copolymer of κ-caragenan [26].

In the work of Jovanovic and Adnadjevic, the isothermal kinetics of the overall crosslinking polymerization of acrylic acid (CPAA) in a CH and MWH reaction process of poly (acrylic acid) (PAA) hydrogel preparation was examined. In the MWH polymerization the reaction rate constant of PAA hydrogel formation increases 32-43 times, when compared to the CH process. The kinetics of the isothermal PAA hydrogel formation during the MWH process was successfully described by the kinetics model of first-order chemical reaction, on the contrary to CH process when the isothermal kinetics is completely described with the second-order chemical reaction rate model. Furthermore, the reaction kinetics of the PAA hydrogel formation in the MWH is changed as well as

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its kinetic parameters. The activation energy (Ea) was decreased ~19% and the pre-exponential factor (lnA) ~2.2 times [27]. Adnadjevic et. al established that the increase in the reaction rate of CPAA for MWH was not caused with the overheating neither with the existence of hot spots in the investigated reaction system. The offered explanation of the effects of MWH on the kinetics of CPAA is based on the specific mode of activation of model of reacting molecules [28]. Nikolic and co-workers presented new synthesis of poly(D,L-lactide) under controlled MWH [29].

In spite of the huge practical significance of the hydrogels as smart materials, the investigations of the kinetics of hydrogel preparation by simultaneous crosslinking polymerization under the isothermal MWH are sparse. There are no works about the synthesis of AA copolymer hydrogels nor the kinetics of FRCCP of the AA acid with MA and AA acid with MAL, under CH and MWH. Therefore, this work investigates for the first time in the literature the effects of methacrylic acid (MA) and maleic acid (MAL) as copolymer units, on the kinetics of the AA copolymerization under CH and MWH.

In regard with that, the aim of this investigation was to determine the kinetics model, kinetics parameters and kinetics complexity and based on them offer the elucidation of the effect of MWH. This paper offers the knowledge about the kinetics of FRCCP under the CH and under the influence of MWH which is important for understanding the kinetics of polymerisation reaction under the MWH and for the advancement of the polymerisation technologies. Furthermore, the knowledge of the kinetics is of crucial importance for the design and projecting scale-up plant.

2. Experimental

2.1 Materials

The monomers, acrylic acid (AA) (99 wt %), methacrylic acid (MA) (99 wt%) and maleic acid (MAl) were purchased from Merck, Germany. The crosslinker, N,N-methylene bisacrylamide (p.a) (MBA) was obtained from Aldrich Chemical, USA. The 2,2-azobis-[2-(2-imidazolin-2-il)-propane] dihydrochloride, which was used as an initiator, was supplied by Wako Pure Chem. Ind., Japan. Double distilled water was used through the all synthesis.

2.2 Hydrogel Synthesis

Both poly(acrylic acid-co-methacrylic acid) hydrogel (PAMA) and poly(acrylic acid-co-methacrylic acid) (PAMAL) hydrogel were synthesized by the free-radical crosslinking co-polymerization of AA, actually using the same procedures. The synthesis of PAMA hydrogel was described in details in our previous papers [30]. The prepared reaction mixture was putted into glass test tubes which were properly sealed and placed in a laboratory oven for predetermined time intervals. After the predetermined time interval, the reaction was stopped by placing test tube with reaction mixture in ice water. Subsequently, the resulting product – formed hydrogel was removed from the test tubes, sliced and immersed in tenfold volume excess of distilled water, in order to remove any sol-fraction. The water is changed each 3 hours, except overnight, during 7 days. Then, the washed-out samples of hydrogels were dried until unchanging weight, by the previously established procedure [30]. Actually, the washed-out hydrogel was dried in an air oven in a temperature regime of 353 K for 2 h, 363 K for 3 h, and 378 K until the sample reaches a constant weight, according to our long-lasting experience the obtained best results. The obtained product (xerogel) was completely dry, opal white solid, and was stored in a desiccator until further use.

Hydrogels synthesis under MWH were performed in a modified microwave unit (Discover, CEM Corp., USA), whose construction and principle of work is described in details in the work of Adnadjevic et al [31].

2.3 Determination of the Yield

The yield (Y, %) of the obtained hydrogel was determined by gravimetric method and calculated as:

$$Y = \frac{W_t}{W_0} \tag{1}$$

where the W_t is the mass of dried washed out hydrogel (xerogel) and the W_o is the mass of the used co-monomers.

2.4 Model-Fitting Method

The kinetics and kinetics models of the investigated reactions were determined by model-fitting method [32]. Detailed description of kinetics models determination based on using experimentally conversion curves is given in our previous works [30,31].

3. Results and Discussion

The conversion curves of PAMA hydrogel (the dependence of the yield on polymerization time) obtained under isothermal conventionally heated conditions at different temperatures are shown in Figure 1.

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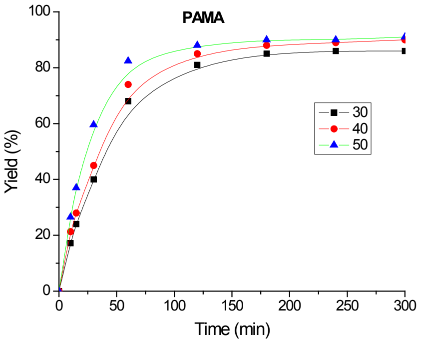

Figure 1. Conversion curves of PAMA hydrogel at CH conditions

The shape of conversion curves of PAMA hydrogel is complex and independent on temperature of polymerization. For all the conversion curves at short reaction times the yield of the obtained hydrogel product rapidly and linearly increases with time, followed with convexly decrease, until attaining the plateau for a long time. The maximal achieved yield of the PAMA hydrogel was ~ 90%. The increasing temperature leads to the increase in both the slope of the linear part of conversion curve and the time to reach plateau decrease. The increase in the value of the slope of linear part of conversion curves and decrease in time required to reach the plateau indicates that the polymerization process is thermally activated one whose rate increase with the increasing temperature.

By using the "model-fitting" method it was concluded that the kinetics curves can be described with the kinetics model of second order chemical reaction, given with Equation (2):

$$Y = \frac{kt}{1 + kt} \tag{2}$$

where the k is the rate constant. In order to calculate the k, the Eq (2) is linearized in the form of dependence of the t/Y on time.

The dependence of t/Y on time of PAMA hydrogel at different temperatures at CH conditions is shown in Figure 2.

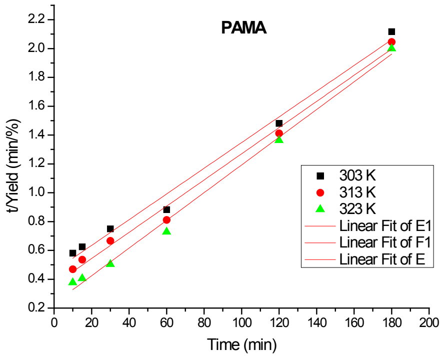

Figure 2. The dependence of t/Y on time for PAMA hydrogel

The dependence of t/Y on time for all of the temperatures provides straight lines for the whole range of the reaction. Since the dependence is straightforward within the entire range, from their slopes are calculated the values of rate constant k for each temperature. Table 1 presents the isothermal values of k for PAMA hydrogel at CH conditions at different temperatures.

Table 1. The values of k for PAMA and PAMAL hydrogel at CH conditions

	PAMA	**PAMAL**
T, K	k, min^{-1}	k, min^{-1}
303	2.19	0.68
313	2.76	0.98
323	4.32	1.28

From the results given in Table 1 it is obvious that the values of k for PAMA hydrogel formation increase with the increaseing reaction temperature. Since the changes in the values of k are in agreement with the Arrhenius Equation, the values of kinetics parameters, activation energy (Ea) and pre-exponential factor (lnA) are calculated by applying it. The calculated values of kinetics parameters are found to be: Ea=27.5 kJ/mol and ln(A/min^{-1})=11.7.

Figure 3 shows the isothermal conversion curves of PAMA hydrogel formation under MWH conditions at different temperatures.

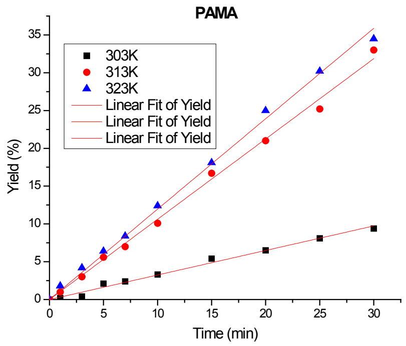

Figure 3. The conversion curves of PAMA at MWH conditions

The conversion curves of PAMA at MWH conditions have different shape than the conversion curves obtained under the CH conditions. The yield of PAMA linearly increase with the increase in duration of polymerization for all the temperatures. The application of the "model-fitting" method indicates that the kinetics curves of PAMA polymerization at MWH conditions should be described by using Polany –Winger model, given with Equation (3):

$$Y = kt \tag{3}$$

Table 2 presents the isothermal values of k for PAMA and PAMAL hydrogel at MWH conditions at different

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temperatures.

Table 2. The values of *k* for PAMA and PAMAL hydrogel at MWH conditions

T, K	PAMA *k*, min^{-1}	PAMAL *k*, min^{-1}
303	0.342	2.242
313	1.035	3.058
323	1.246	3.756

The results given in Table 2 reveals that the values of the *k* for PAMA hydrogel at MWH conditions increasing with the reaction temperature in compliance with Arrhenius equation, as in previous case of CH conditions. The kinetics parameters values, activation energy and pre-exponential factor, for PAMA at MWH conditions were calculated as following: Ea=50.1 kJ/mol and ln(A/min^{-1}) = 20.9.

By comparing kinetics data for PAMA hydrogel at CH and MWH conditions one can conclude the following. The MWH most probably provokes the alteration in the kinetics model of FRCCP of AA and MA, the decrease in the value of the *k* and leads to the increase in the values of kinetics parameters. A functional relationship between the kinetics parameters for CH and MWH conditions is found and is expressed with following Equation:

$$\ln A = 0.471 + 0.408 E_a \tag{4}$$

where $E_{a,F}$ and $\ln A_F$ are the activation energy and pre-exponential factor in a defined physical field [33]. By its form, Equation (4) is well-known as the Equation of the compensation effect, which is related to variations in the reactions settings.

By comparing kinetics data obtained for PAMA hydrogel with the results obtained for PAA hydrogel [28] it is clear that the introduce of methacrylic acid (MA) as co-monomer does not change the kinetics model, but provokes the decrease in the rate of polymerization and the increase in kinetic parameters, both under CH and MWH conditions.

The isothermal conversion curves of PAMAL hydrogel obtained under CH conditions at different temperatures are given in Figure 4.

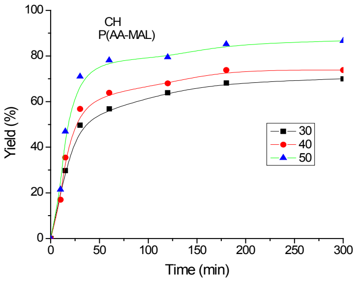

Figure 4. Conversion curves of PAMAL hydrogel at CH conditions

The conversion curves of PAMAL hydrogel have comparable forms for all experimental temperatures, showing the 3 distinct stages of the changes of the yield of the PAMAL hydrogel formation with time: a linear, non-linear (convexly) and plateau. As in the case of PAMA hydrogel, with the increase in temperature of polymerization the maximal yield of PAMAL hydrogel increase. At the start of the process, i.e. at short reaction times, the yield of the PAMAL hydrogel linearly and abruptly increases with time, after which slowed down, until reaching saturation at long limes. The slope of the linear part of the conversion curves increase with the increase in temperature, whereas the time to reach plateau decreases. As in the case of the PAMA hydrogel formation, these facts indicate that investigated FRCCP is thermally activated process.

The application of the "model-fitting" method reveals that the kinetics curves can be described by using the Equation (2), as in the case of PAMA. In accordance with that, the dependence of t/Y on time for PAMAL at CH conditions at different temperatures is investigated and presented in Figure 5.

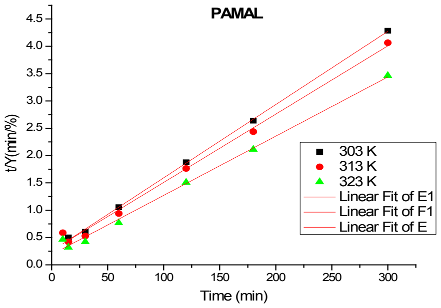

Figure 5. The dependence of t/Y on time for PAMAL

As obvious from Figure 5, the above dependency is straight within the whole range at all the temperatures. The values of the *k* are calculated based on the slopes of these dependencies. The calculated isothermal values of *k* for PAMAL formation at different temperatures at CH conditions are given in Table 1 (column 3). The increasing values of *k* with temperature are in agreement with the Arrhenius Equation, which allows calculation of the values of kinetics parameters, activation energy (Ea) and pre-exponential factor (lnA). The calculated values of the kinetics parameters are: Ea=24.9 kJ/mol and ln(A/min^{-1})=9.5.

Figure 6 shows the isothermal conversion curves of PAMAL hydrogel obtained under MWH conditions at different temperatures.

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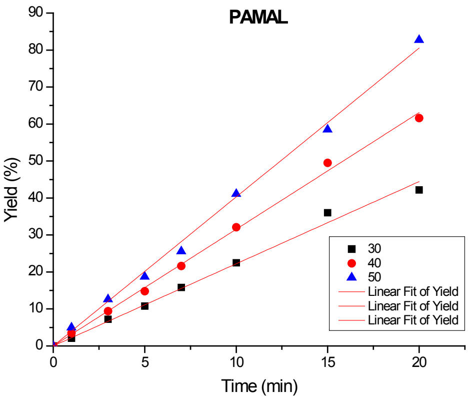

Figure 6. Conversion curves of PAMAL at MWH conditions

Similarly to the previously presented results for the PAMA at MWH conditions, the conversion curves are obviously linear, within the whole investigated range of PAMAL polymerization reaction. The dependence of the yield on reaction time for PAMAL at MWH can be described with the Equation (3).

The slope of the conversion curves, again, increase with the increase in reaction temperature, and from their values the values of *k* for PAMAL at MWH conditions at different temperatures are calculated (see Table 2). As in the previously explained cases, the values of kinetics parameters, Ea and lnA at MWH conditions are calculated and found to be: Ea= 21.3kJ/mol and $\ln(A/\text{min}^{-1})=9.2$.

As in the case of the use of MA as co-monomer, the kinetics model of PAMAL polymerization under the MWH conditions is found to be different than under the CH conditions. On the contrary to the MA, in the case of PAMAL the MWH leads to the increasing rate constants values and to the decreasing kinetics parameters values in comparison to their values obtained under the CH conditions. Between the kinetics parameters of PAMAL at CH and MWH conditions it is found the existence of correlation relationship - compensation effect, given with following equation:

$$\ln A = 6.911 + 0.106 E_a \tag{5}$$

The results presented in this manuscript enable to make comparison between the two types of co-monomers and their effects in the kinetics of the FRCCP under both CH and MWH conditions. On the other side, it is possible to discuss the effects of heating mode, CH or MWH on the kinetics of the investigated isothermal process. Under the CH conditions, it is possible to successfully describe the kinetics of both PAMA and PAMAL hydrogel formation using same kinetics model, i.e. by the kinetics model of second order chemical reaction. The values of kinetics parameters are higher for PAMA. Under the MWH conditions, the kinetics of both PAMA and PAMAL hydrogel formation again can be successfully described by using the same kinetics model but different than in the case of the same processes under CH conditions, i.e. by Polany-Winger model. The values of kinetics parameters are higher for PAMA.

In the works of Adnadjevic, Jovanovic, et al, the so called "model of selective energy transfer (SET)" of activation of reactive molecules is presented, with aim to explain the effects of MWH on the kinetics of physicochemical processes such as polymerization and dehydration [9,19,27,28,30,31]. In agreement with that model, in the case when exists compensation effect between the calculated values of kinetics parameters obtained under different modes of heating of reaction system, it is possible to calculate the values of: the anharmonicity constant (x), resonant vibration mod of monomers molecule (v) and the vibration quantum number (n). The calculated values of v, n and x for PAMA and PAMAL hydrogel formation under MWH and CH are given in Table 3.

Table 3. The values of v, n, and x for PAMA and PAMAL hydrogel formation

	PAMA		**PAMAL**	
Variable	CH	MW	CH	MW
v /cm^{-1}	410	410	1579	1579
n	6	10	2	1
x	-0.025	-3.45×10^{-3}	-0.204	-7.9×10^{-3}

From the results given in Table 3, it is indicating that in the case of PAMA hydrogel formation, both under CH and MWH, the resonant transfer of energy takes places at vibration mode with wave number $v = 410$ cm^{-1}, which matches to the skeletal vibrations of the monomer unit -C-C-C-C- of AA and MA. In contrast to this, in the case of PAMAL resonant transfer of energy takes places at vibration mode with wave number $v = 1579$ cm^{-1}, which corresponds to stretching asymmetric vibrations of carboxylic group C-O-O of the monomer unit of AA and MAL.

The increase in the value of Ea and lnA under MWH in comparison with the CH in the case of PAMA hydrogel is related with the quant nature of activation energy and can be explained with the higher number of the necessary quant of energy and with the higher value of the anharmonicity factor. That increase happened due to the specific interaction of MW irradiation with the molecules of methacrylic acid. The decrease in the value of Ea and lnA under MWH in the case of PAMAL hydrogel is also relat-

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ed with the quant nature of activation energy and can be explained with the lower number of the necessary quant of energy and with the lower value of the anharmonicity factor. That decrease happened due to the specific interaction of MW irradiation with the molecules of maleic acid.

4. Conclusions

(1) The kinetics of FRCCP of PAMA and PAMAL hydrogel under the CH conditions is described with the kinetics model of second order chemical reaction.

(2) The kinetics of FRCCP of PAMA and PAMAL hydrogel under the MWH conditions is described with the kinetics model of Polany-Winger.

(3) The type of co-monomer does not affect the kinetics model within the same heating mode.

(4) The MWH provokes the alteration in the kinetics model of crosslinking co-polymerization for both commoners in comparison to the CH.

(5) The type of commoner effects on the values of kinetics parameters for both CH and MWH.

(6) The values of Ea and lnA are under the conditions of MWH in comparison to the CH are higher for PAMA and lower for PAMAL.

(7) The relationships among the kinetic parameters values for MWH and CH conditions for both FRCCP are present (compensation effect).

(8) The resonant transfer of energy during crosslinking co-polymerization for PAMA formation takes places at vibration mod with wave number $v = 410\ cm^{-1}$, both under CH and MWH, which corresponds to the skeletal vibrations of the monomer unit -C-C-C- of AA and MA. In contrast to this, in the case of PAMAL hydrogel, resonant transfer of energy takes places at wave number $v = 1579\ cm^{-1}$, which corresponds to stretching asymmetric vibrations of carboxylic group C-O-O of the monomer unit of AA and MAL.

(9) The activation energy for both co-monomers is quantized value and it is determined with the number of quanta exchanged throughout the selective transfer of energy.

(10) The application of MWH opens wide perspectives for designing novel, cost effective technologies for products based on polymers.

(11) The application of SET model mechanism of activation leads to the development of novel polymerization reaction systems.

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