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KINETICS OF CARBOXYMETHYLATION REACTION OF CELLULOSE SAMPLES WITH DIFFERENT DEGREE OF CRYSTALLINITY

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The aim of this work is to study the kinetics of the carboxymethylation reaction of samples of cotton cellulose (CC), microcrystalline cellulose (MCC), and powdered cellulose (PC) with significantly different values of the degree of crystallinity by solid-phase and suspension methods under the same adiabatic conditions. The dependence of the degree of substitution of the obtained samples of carboxymethyl cellulose (CMC) on the rate of the carboxymethylation reaction, the rate constant, and the duration of the reaction was studied. Based on the Arrhenius equation, the thermal effect of the carboxymethylation reaction, the activation energy of the reaction, exponential values, and the reactivity of the reagents were studied.

Keywords: carboxymethylcellulose, cotton cellulose, powdered cellulose, microcrystalline cellulose, reaction rate, activation energy, thermal effect, kinetics

КИНЕТИКА РЕАКЦИИ КАРБОКСИМЕТИЛИРОВАНИЯ ОБРАЗЦОВ ЦЕЛЛЮЛОЗЫ РАЗЛИЧНОЙ СТЕПЕНИ КРИСТАЛЛИЧНОСТИ

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Целью работы является исследование кинетики реакции карбоксиметилирования образцов хлопковой целлюлозы (ХЦ), микрокристаллической целлюлозы (МКЦ) и порошкообразной целлюлозы (ПЦ) с существенно отличающимися значениями степени кристалличности твердофазным и суспензионным методами при одинаковых адиабатических условиях. Исследована зависимость степени замещения полученных образцов карбоксиметилцеллюлозы (КМЦ) от скорости реакции карбоксиметилирования, константы скорости, продолжительности реакции. На основе уравнения Аррениуса изучены тепловой эффект реакции карбоксиметилирования, энергия активации реакции, экспоненциальные величины и реакционная активность реагентов.

Ключевые слова: карбоксиметилцеллюлоза, хлопковая целлюлоза, порошковая целлюлоза, микрокристаллическая целлюлоза, скорость реакции, энергия активации, тепловой эффект, кинетика

TURLI KRISTALLANISH DARAJALI SELLULOZA NAMUNALARINI KARBOKSIMETILLASH REAKSIYASI KINETIKASI

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Ushbu ishning maqsadi bir xil adiabatik sharoitda qattiq faza va suspenziya usullari bilan kristallik darajasining sezilarli darajada farq qiladigan qiymatlari bo'lgan paxta tsellyulozasi (PTs), mikrokristalik tsellyuloza (MKTs) va kukunsimon tsellyuloza (KTs) namunalarining karboksimetilatsiya reaktsiyasining kinetikasini o'rganishdan iborat. Olingan karboksimetiltsellyuloza (KMTs) namunalarini almashtirish darajasining karboksimetillanish reaktsiyasi tezligiga, tezlik konstantasiga va reaksiya davomiyligiga bog'liqligi o'rganildi. Arrenius tenglamasi asosida karboksimetillanish reaksiyasining issiqlik effekti, reaksiyaning faollanish energiyasi, eksponensial qiymatlari va reagentlarning reaktsion faolligi o'rganildi.

Kalit so'zlar: karboksimetilselluloza, paxta sellulozasi, mikrokristallik selluloza, kukunsimon selluloza, reaksiya tezligi, faollanish energiyasi, issiqlik effekti, kinetika

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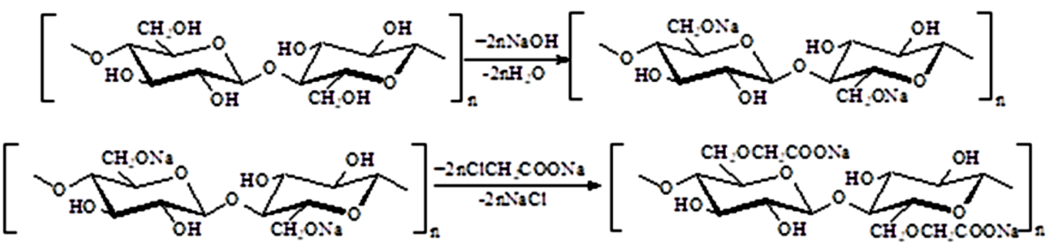
Kirish

KMS-sellulozaning keng tarqalgan oddiy efiri hisoblanib, sanoatning turli sohalarida, jumladan, neft-gaz [1], qurilish [2], to'qimachilik [3], qishloq xo'jaligi [4], qog'oz sanoatlarida [5] va bo'yoq ishlab chiqarish [6], rudalarni boyitish[7], shuningdek, uning tozalangan markalari oziq-ovqat [8], tibbiyot [9], farmatsevtika [10], parfyumeriya [11] va boshqa sohalarda keng qo'llaniladi [12].

KMS olishda turli selluloza tutuvchi xomashyolar yog'och o'simliklar sellulozasi [13], MKS [14], lignotselluloza biomassasidan [15], Tithonia o'simligining poyasi va bargi [16], pishmagan mevasidan [17], paxta chiqindilari [18], qog'oz chiqindilari [19], tekstil chiqindilari [20], paxta sellulozasi [21], makkajo'xori boshoqlari [22], banan po'stlog'i [23], apelsin qobig'i [24], shakarqamish poyasi [25], palma mevasi [26], kakao po'stlog'i [27], makkajo'xori qobig'i [28], makkajo'xori poyalari [29], Asparagus officinalis poyalari [30], Eucalyptus globulus [31], trikotaj chiqindilari [32] asosida turli sifat ko'rsatkichlarga ega bo'lgan KMS namunalari olingan.

KMS namunalarini olish jarayoni ikki bosqichli kimyoviy reaksiya orqali amalga oshirildi. Birinchi bosqichda selluloza namunalari natriy gidroksid eritmasi bilan ishlov berildi. Ikkinchi bosqichda, hosil bo'lgan ishqoriy selluloza alkillovchi agent ishtirokida karboksimetillash reaksiyasi amalga oshirildi [33]:



Ushbu reaksiya bilan bir qatorda alkillovchi agentning gidrolizi hisobiga quyidagi qo'shimcha reaksiyalar ham sodir bo'ladi [34]:

$$ClCH_2COONa + H_2O \longrightarrow OHCH_2COOH + NaCl$$

etanol-2 kislota

$$OHCH_2COOH + NaOH \longrightarrow OHCH_2COONa + H_2O$$

2-etankislotaning natriyli tuzi

$$ClCH_2COONa + NaOH \longrightarrow OHCH_2COONa + NaCl$$

Ushbu qo'shimcha reaksiyalar hisobiga oxirgi mahsulot tarkibida 50 % gacha qo'shimcha noorganik tuzlar hosil bo'ladi. Reaksiya sharoiti, sintez qilish usuli, xomashyo turi, ishlab chiqarish texnologiyasini tanlash orqali asosiy reaksiyaning samaradorligini oshirishga erishiladi. Karboksimetillash reaksiyasi unumi, reaksiya tezligi, mahsulotning sifat ko'rsatkichlari selluloza tutuvchi dastlabki xomashyo ustmolekulyar tuzilishi va boshqa ko'rsatkichlariga bog'liq bo'ladi. Ushbu tadqiqotda bir xil sharoitda turli ustmolekulyar tuzilishga ega bo'lgan PS, KS va MKS namunalarini qattiq fazali va suspenzion usullarda karboksimetillash reaksiyasi kinetikasi bir-biriga taqqoslangan holda tadqiq qilindi.

Tadqiqot usullari

Tajribani o'tkazish uchun zarur bo'lgan kimyoviy modda va reaktivlar:

- paxta sellyulozasidan(GOST 595);
- natriy gidroksid (GOST 4328-68) kvalifika -tsiyasi “a.u.t”;
- monoxlorsirka kislota (MXSK) “imp”;
- texnik etil spirt (GOST 17299) kvalifika-tsiyasi “k.t.”;
- distillangan suv (GOST 6709).

5 g sellyulozaga 30 ml 70 % li etil spirt eritmasi bilan 15 minut vaqt davomida ishlov beriladi. Olingan massa 20 ml 40 % li natriy gidroksid eritmasi bilan aralashtiriladi. Sellyulozani ishqor bilan ishlov berish jarayoni 16 ^{o}C xaroratda 90 minuntgacha davom ettiriladi. So'ngra olingan reaksion aralashma bilan monoxlorsirka kislota eritmasi o`zaro 55 ^{o}C xaroratda 3 saot davomida aralashtiriladi. Olingan mahsulot filtrlanadi va 60-70 ^{o}C xaroratda doimiy massaga kelguncha quritiladi [35].

KMS namunalarining AD qiymati [36] Ts 19515439-01:2017 bo’yicha xisoblab topiladi.

*Karboksimetillash reaksiyasi kinetikasini aniqlash***.** Karboksimetillash reaksiyasining tezlik konstantasi AD bog'liq xolatda aniqlanadi:

$$K = \frac{d\gamma}{d\tau(\gamma_{max}-\gamma)} \qquad (3)$$

Bu yerda: γ – almashinish darajasi; γ_{mak} – almashinish darasining umumiy miqdori.

Karboksimetillash reaksiyasining tezligi Arrenius tenglamasi bilan xisoblanadi [37]:

$$k = A \cdot e^{-\left(\frac{E_A}{RT}\right)} \qquad (4)$$

Bu yerda: A – reaksiya komponentlarining tanlangan molyar nisbati; E – aktivlanish energiyasi, J/mol; R – universal gaz doimiysi, J/mol·K; T – xarorat, K.

Karboksimetillash reaksiyasida qatnashgan PS, MKS, KS namunalarining aktivlanish energiyalarini quyidagi formula asosida hisoblab topiladi:

$$E = tg\alpha \cdot R \qquad (5)$$

Karboksimetillash reaksiyasining eksponensial qiymatlari quyidagi formula asosida hisoblab topiladi:

$$A = \exp(\ln A) \qquad (6)$$

Eksponensial qiymatlar asosida reaksiyaning issiqlik effekti qiymatlari quyidagi formula asosida hisoblab topiladi:

$$Q = cmd\tau \qquad (7)$$

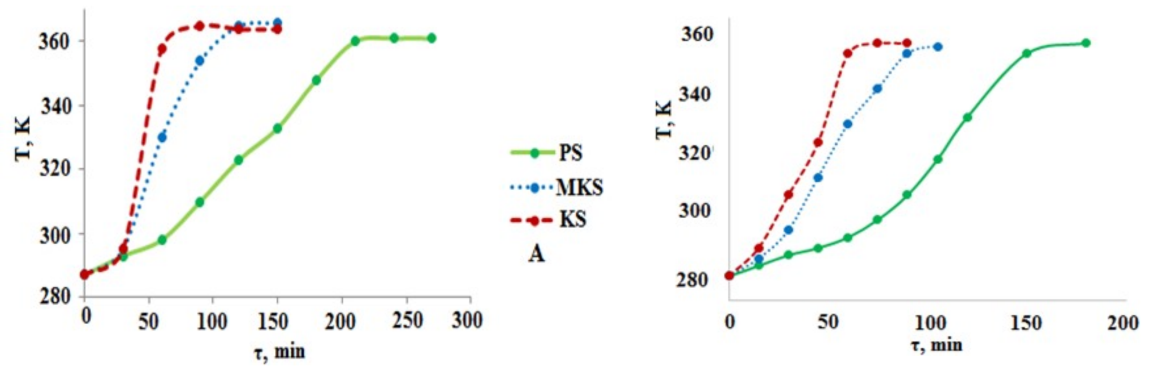
Sellulozani karboksimetillash reaktsiyasi SN1 va SN2 reaksiya mexanizmining oraliq xolatini egallaydi. Bunlay karboksimetillash reaktsiyasining kimyoviy kinetikasi quyidagi formula orqali ifodalaniladi [38].

$$\frac{dAD}{d\tau} = k(AD_{max} - AD) \qquad (8)$$

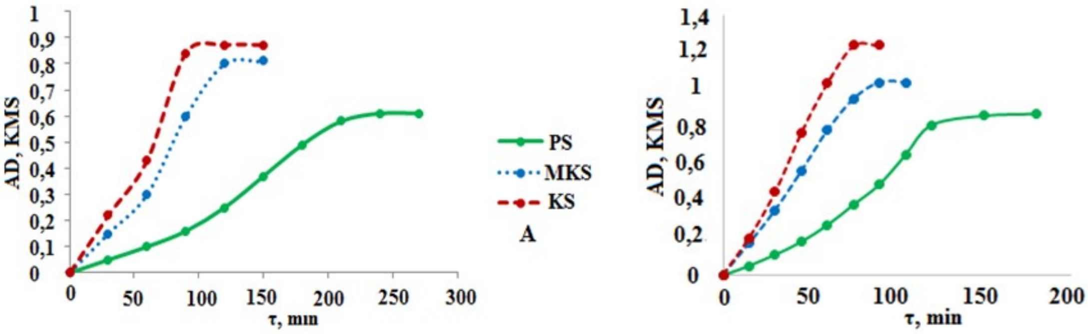
Bu yerda: k- reaksiyaning differensial orqali olingan tezlik konstantasi, 1/c; AD – KMS namunalarining almashinish darajasi (massa ulushda); AD_{max} – eng yuqori almashinish darajasi qiymati.

Natijalar va muhokama

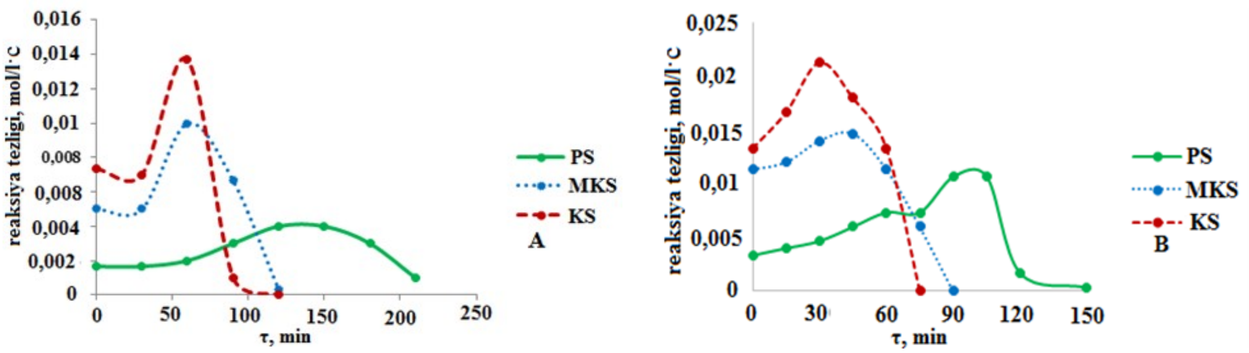
Oldingi tadqiqotlarimizda turli sellyuloza saqlovchi xomashyolardan qattiq fazali va suspenzion usullarda suvda to'liq eruvchan, yuqori va quyi ADli KMS namunalari sintez qilishning optimal sharoitlari aniqlangan[39]. Bunda PS, KS, va MKS asosida AD qiymati 0,38-0,55 bo'lgan suvda to'liq eruvchan quyi ADli KMS namunalari olishga erishilgan [40]. Olib borilgan tadqiqotlar shuni ko'rsatadiki, bir xil sharoitda turli selluloza namunalarini karboksimetillash jarayonida har xil sifat ko'rsatkichlarga ega bo'lgan KMS namunalari hosil bo'ladi. Ushbu tadqiqot ishida, adiabatik sharoitda, ustmolekulyar tuzilishi va kristallanish darajasi jihatdan bir biridan keskin farq qiluvchi PS, KS va MKS namunalarini karboksimetillash reaksion faol-



1-rasm. A-qattiq B-suspenzion usulda karboksimetillash reaksiya vaqtining haroratga bog'liqligi.



2-rasm. A-qattiq B-suspenzion usulda olingan KMS almashinish darajasining vaqtga bog`liqligi.



3-rasm. A-qattiq fazali B-suspenzion usullarda karboksimetillash reaksiyasi tezligining dАД/dτ=f(τ) karboksimetillash vaqtiga bog`liqligi(1/c).

liklarini baholash bo'yicha olingan natijalar o'rganildi. Bunda, suspenzion usul va qattiq fazada PS, KS va MKS namunalarini karboksimetillash reaksiyasi kinetikasi, reaksiya tezligi, xarorat, vaqt, reaksiyaning tezlik konstantasi, faollanish energiyasi, jarayonning issiqlik samaradorligi va boshqa ko'rsatkichlarning o'zaro bog'liqligi taqqoslab tadqiq etildi.

Karboksimetillash reaksiyasi maxsus dyuardan iborat bo'lgan qurilmada, adiabatik sharoitda olib borildi va reaksiya samaradorligi vaqt bo'yicha harorat hamda maxsulotning AD qiymati o'zgarishi orqali aniqlandi (1-rasm).

Qattiq fazada 360 K haroratda PS KS va MKS namunalarini karboksimetillash reaksiyasi uchun sarflangan vaqt mos ravishda 270, 150, 150 minutni tashkil qildi. Suspenzion usulda organik erituvchi muhitida 350 K haroratda PS, KS va MKS namunalarini karboksimetillash reaksiyasi uchun tegishlicha 200, 100, 120 minut vaqt sarflandi.

Qattiq fazali va suspenzion sharoitda olingan KMS ADsining karboksimetillash reaksiyasi vaqtiga bog'liqligi o'rganildi. AD= (2-rasm).

Qattiq fazada karboksimetillash reaksiyasi PS uchun 270 minut, MKS, KS uchun esa 150 minutga yetganida AD mos ravishda 0,6; 0,8 va 0,9 suvda to'liq eruvchan KMS namunalari sintez qilindi. Suspenzion usulda PS, KS, MKS namunalarini karboksimetillash reaksiyasi tegishlicha 180, 100 va 120 minutda AD 0,8, 1,2, 1 bo'lgan KMS namunalari sintez qilindi.

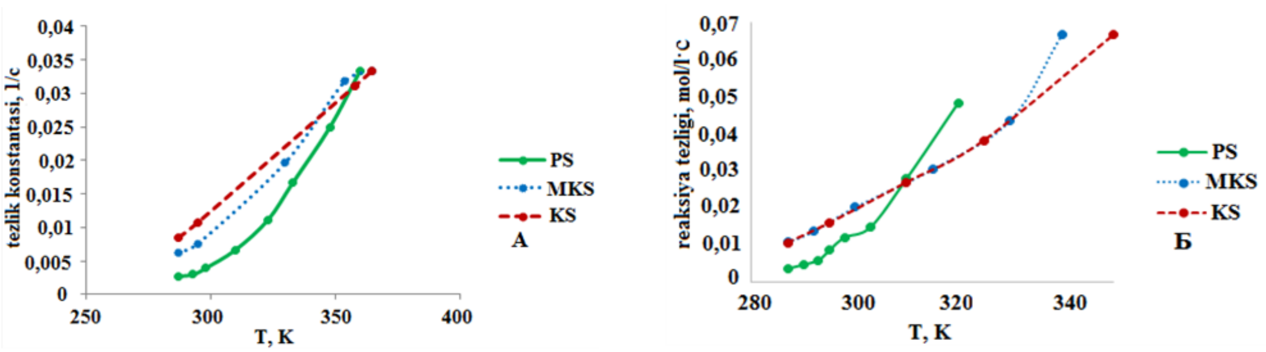
Karboksimetillash reaksiyasi vaqti k=f(τ) bilan karboksimetillash reaksiyasi tezligining (dγ/dτ) o'zaro bog'liqligi o'rganildi (3-rasm).

Qattiq fazada PS va MKS, KS namunalarini karboksimetillash reaksiyasining tezlik konstantalari mos ravishda 210 va 120 minut vaqt oraliqlarida yuqori qiymatlarga ega bo'ldi. Suspenzion usulda karboksimetillash reaksiyasining tezlik konstantalari PS, MKS, KS uchun tegishlicha 150, 90, 70 minutda yuqori qiymatlarda bo'lgan. Belgilangan vaqt oraliqlarida qattiq fazali va suspenzion usullarda karboksimetillash reaksiyalarining tezligi yuqori qiymatlarda bo'lgan.

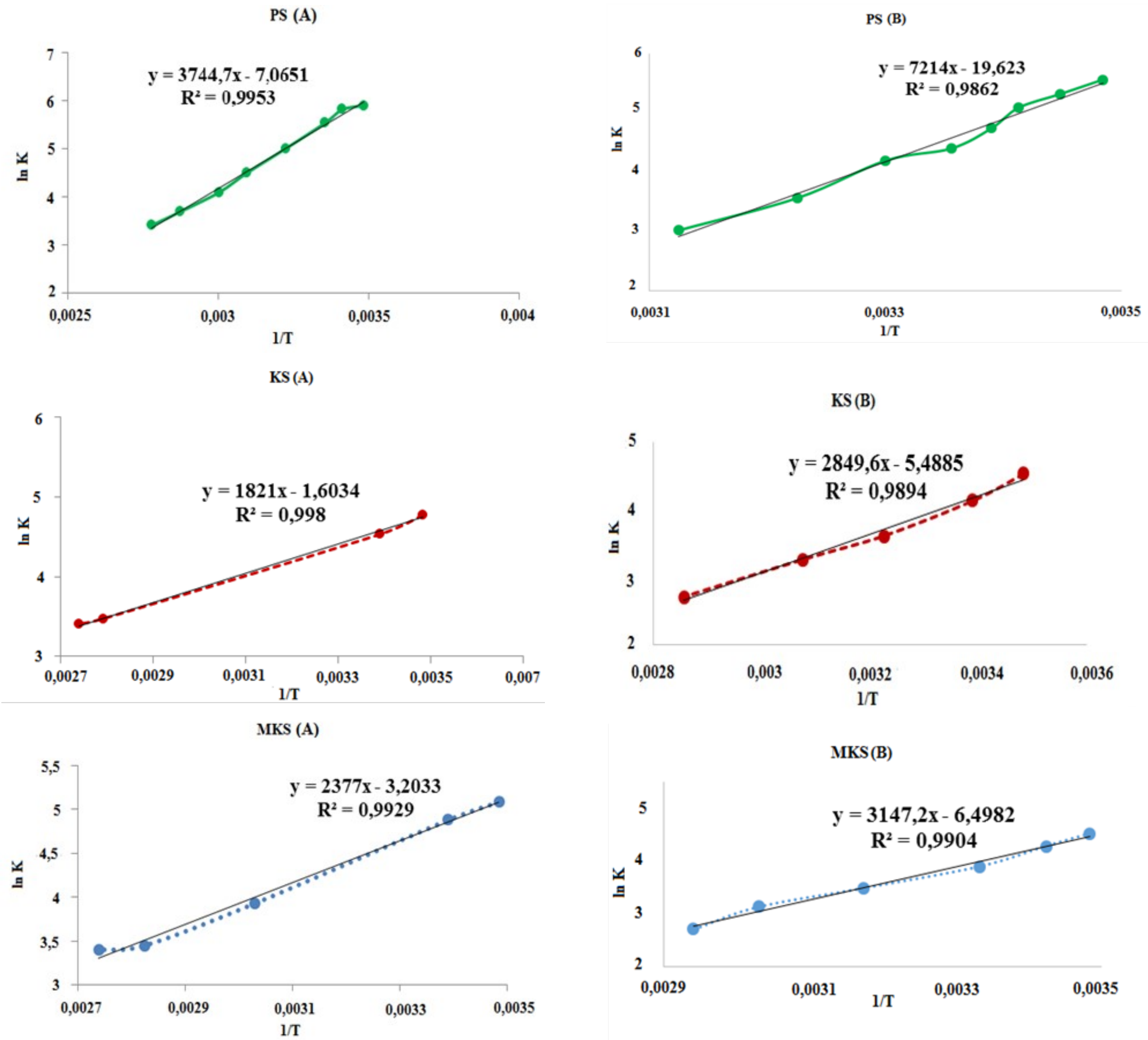
PS, MKS, KS namunalarining tezlik konstantasi (K) bilan karboksimetillash reaksiyasi xaroratining o'zaro bog'liqligi o'rganildi (4-rasm).

Qattiq fazada karboksimetillash reaksiyasining tezlik konstantasi 350 K, suspenzion usulda esa PS, MKS, KS namunalari uchun 320 K, 330 K, 350 K haroratlarda eng yuqori qiymatga ega bo'lishi aniqlandi.

PS, KS, MKS karboksimetillash reaksiyalarining aktivlanish energiyalari E=tgα·R formula



4- rasm. A-qattiq fazali B-suspenzion usullarda karboksimetillash reaksiyasi tezlik konstantasining xaroratga bog'liqligi k=f(τ).



5- rasm. A-qattiq fazali B-suspenzion usullarda karboksimetillash reaksiyasining differensial 1/T bilan lnK bog'liqligi.

PS, KS va MKS namunalarining karboksimetillanish reaksiyasining kinetik parametrlari

Namuna	Qattiq fazada olingan			Suspenzion fazada olingan		
	E, J/mol	A, 1/c	Q_r, кJ/mol	E, J/mol	A, 1/c	Q_r, J/mol
PS	31118	3179	1091	59948	3321,0	1221
MKS	19753	24,6	1797	26153	663,5	1985
KS	15722	6,07	1905	23680	241,7	2160

bilan hisoblash uchun - lnK va 1/T formulaning bog'liqligi asosida tgα qiymati aniqlandi (5-rasm).

Qattiq fazali va suspenzion usullarda PS, MKS, KS namunalarini karboksimetillash reaksiyasining faollanish energiyalari (E=tgα·R), eksponensial koeffitsient (A=exp (lnA)), reaksiyasining issiqlik effekti qiymatlari aniqlandi va quyidagi jadvalda keltirildi (jadval).

Qattiq fazali va suspenzion usulda karboksimetillash reaksiyasi natijasida eksponensial va kimyoviy faollanish energiyalari qiymati kamayib, reaksiyaning issiqlik effekti qiymati ortib borishi PS, MKS, KS namunalarining ustmolekulyar tuzilishi bilan bog'ligi ko'rsatildi. Selluloza saqlovchi xomashyolarning kimyoviy tuzilishi karboksimetillash reaksiyasining tezligiga, tezlik konstanlariga, va reaksiyaning kinetik parametrlariga ta'sir qilishi ko'rsatildi.

Xulosa

Turli kristallanish darajasiga ega PS, MKS, KS namunalarini reaksion faolligi ularning ustmolekulyar tuzilishiga bog'liq ekanligi aniqlandi. PS, MKS, KS namunalarini karboksimetillash reaksiyasining tezlik konstantasi bilan xarorat o'zaro to'g'ri proporsional ekanligi aniqlandi. Turli kristallanish darajalariga ega bo'lgan selluloza saqlovchi namunalarni karboksimetillash reaksiyasining aktivlanish energiyalari, reaksiyaning faollanish enegiyasi, turli xaroratlarda reaksiyaning tezlik konstantalari va issiqlik effektlari aniqlandi.

O'zbekiston Respublikasi Innavatsion rivojlanish vazirligining 2022-2023-yillarga mo'ljallangan O'zbekiston-Belorussiya № MRB 2021-548 "Turli funksional maqsadlar uchun organik va noorganik qoplamali modifikatsiyalangan tolali materiallarni yaratish" xalqaro ilmiy loyihasi doirasida bajarilgan.

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