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SYNTHESIS OF POLYMETHYLENE NAPHTHALENE SULFONIC ACID BASED ON THE SECONDARY PRODUCT OF THE PYROLYSIS PROCESS

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Purpose of scientific research - the need for the synthesis and study of physico-chemical properties of polymethylennaphthalene sulfocyanate on the basis of naphthalene fraction obtained on the basis of fractional driving of pyrolysis process secondary yield "pyrolysis oil"is due be using that the use of secondary products instead of imported superplasticizers and cationites used in construction and manufacturing industrial enterprises. The conditions of the process of synthesis of linear and spatial polymethylennaphthalene sulfocyanate using naphthalene fraction obtained by fractional rectification of pyrolysis secondary product "pyrolysis oil" were studied. The structures of resulting new superplasticizer (SP) and cationite (ASO) were studied using IR spectroscopy and scanning electronic microscopy methods. The thermal analysis of cation exchangers was performed. The effect of superplasticizer on concrete mixtures has been studied. The operational characteristics of cationite have been determined, as SEC (static exchange capacitance) - 4,6 mg-ekv/g and DEC (dynamic exchange capacitance) - 475-490 mole/m^3. It was found that the addition of 0,8% superplasticizer has increased the strength of concrete on 84,39%.

Keywords: used pyrolysis oil, naphthalene, polycondensation, sulfanation, polymethylennaphthalene sulfocyanate, superplasticizer, sulfocationite, thermal and chemical stability, exchange capacity

СИНТЕЗ ПОЛИМЕТИЛЕННАФТАЛИН СУЛЬФОКИСЛОТ НА ОСНОВЕ ВТОРИЧНОГО ПРОДУКТА ПРОЦЕССА ПИРОЛИЗА

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Цель исследования - синтез и изучение физико-химических свойств полиметиленнафталин сульфокислот на основе нафталиновой фракции, получаемой фракционной перегонкой вторичного продукта процесса пиролиза "пиролизное масло", для замены импортных суперпластификаторов и катионитов, эффективно применяемых на строительных и промышленных предприятиях. Исследованы условия процесса линейного и пространственного синтеза полиметиленнафталин сульфокислоты с использованием фракции нафталина, полученного фракционной перегонкой вторичного продукта пиролиза "пиролизное масло". Структура полученного нового суперпластификатора (СП) и катионита (АСО) изучалась методами ИК спектроскопии и сканирующий электронный микроскопией. Проведено исследование термических свойств катионита, изучено влияние суперпластификатора на бетонные смеси. Определены эксплуатационные свойства катионитов, такие как СОЕ (статическая обменная емкость) 4,6 мг-экв/г и ДОЕ (динамическая обменная емкость) 475-490 моль/м^3. Установлено, что добавление 0,8% суперпластификатора повышает прочность бетона на 84,39%.

Ключевые слова: пиролизное масло, нафталин, поликонденсация, сульфирование, полиметиленнафталин сульфокислота, суперпластификатор, сульфокатионит, термическая и химическая стабильность, обменная емкость

PIROLIZ JARAYONI IKKILAMCHI MAHSULOTI ASOSIDA POLIMETILENNAFTALIN SULFOKISLOTA SINTEZI

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Tadqiqot maqsadi – pirolizi jarayoni ikkilamchi mahsuloti "piroliz moyi"ni fraksion haydash natijasida olingan naftalin fraksiyasi asosida polimetilennaftalin sulfokislota sintez qilish va fizik-kimyoviy xossalarini aniqlash, ikkilamchi mahsulotlardan samarali foydalangan holda qurilish va ishlab chiqarish sanoat korxonalarida ishlatiladigan import superplastifikator va kationitlar sifatida foydalanish. Piroliz ikkilamchi mahsuloti "piroliz moyi"ni fraksion haydash orqali olingan naftalin fraksiyasidan foydalangan holda chiziqli va fazoviy polimetilennaftalin sulfokislota sintezi jarayoni sharoitlari o'rganildi. Olingan yangi superplastifikator(SP) va kationitning(ASO) tuzilishi IQ spektroskopiya va SEM(skanerlovchi elektron mikroskop) usullari yordamida o'rganildi. Kationitning TGA/DTA termal tahlili o'tkazildi. Superplastifikatorni beton aralashmalariga ta'siri o'rganildi. Kationitlarning eksplutatsion xossalari aniqlandi. Kationitlarning muhim ekspluatatsion xossasi COE (statik almashinish sig'imi) va DOE (dinamik almashinish sig'imi) aniqlandi. COE = 4,6 mg-ekv/g, DOE = 475-490 mol/m^3. 0,8% superplastifikator qo'shilganda beton mustahkamligini 84,39% ga oshirishi aniqlandi.

Kalit so'zlar: piroliz moyi, naftalin, polikondensatsiya, sulfolash, polimetilennaftalin sulfokislota, superplastifikator, sulfokationit, termik va kimyoviy barqarorlik, almashinish sig'imi

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Kirish

Hozirgi vaqtda qurilish sanoati juda tez sur'atlar bilan rivojlanib bormoqda. Qurilish sanoatida xomashyo va energiya resurslaridan oqilona va samarali foydalanishga qo'yiladigan talablar ham mos ravishda o'zgarib, betonning mustahkamligi, ishonchliligi va chidamliligi yuqori bo'lgan yig'ma, monolit beton va temir-beton konstruksiyalarni ishlab chiqarish muammosini samarali hal qilish uchun maxsus kimyoviy qo'shimchalardan keng foydalanishni talab qiladi [1]. Piroliz moyi tarkibida naftalinning ko'p miqdorda bo'lishi tufayli undan foydalanishning muhim yo'nalishlaridan biri beton uchun plastifikatorlar ishlab chiqarish hisoblanadi. Beton materiallari mustahkamligini kamaytirmasdan oquvchanligini oshirishda superplastifikatorlar — polimer qo'shimchalar keng qo'llaniladi. Kimyoviy plastifikatorlar tarkibi jihatidan naftalin sulfokislotasining formaldegid kondensati, melamin formaldegid kondensati, lignosulfonatlarning modifikatsiyasidan olingan maxsulotlardan iborat [2, 3]. Hozirgi kunda dunyo bo'yicha yiliga 1,25 mln tonnadan ortiq superplastifikatorlar ishlab chiqarilmoqda. Bu ko'rsatgich yildan-yilga ortib borayapti. Bir qancha C-3, SMF, Dofen DF, Kratasol, Superplast, Polyplast, Ferrokrit, Vilakom, Rheobuild 2000 (Rossiya); Agiplast (Rhona, Fransiya); Cormix (Rhodia, BuyukBritaniya); Chriso fluid (Chriso Industries, AQSh) kabi superplastifikatorlarning asosiy tarkibi polimetilennaftalin sulfokislotasi asosida ishlab chiqariladi [4-6].

Kimyo sanoatni esa sintetik ionitlarsiz ta'savvur qilib bo'lmaydi chunki, ionitlar turli sohalarda ishlatiladi: tozalangan yoki tuzsizlantirilgan suv olishda, gidrometallurgiya sanoatida rangli va qimmatbaho metallarni ajratishda, oqova suvlardan toksik va og'ir metallarni ajratish uchun ishlatilib kelinmoqda [7-10]. Bundan tashqari so'ngi yillarda radiaktiv elementlarni o'z ichiga olgan suvlarni ionitlar yordamida tozalash yoki radiaktiv izotoplarni ajratib olishda qo'llanilmoqda. Shuni takidlash kerakki, stirol va divinilbenzol sopolimerlari asosida olingan ionitlar radiaktiv chiqindi suvlarni tozalashda qo'llanilmaydi. Chunki, bu ionitlar radiaktiv nurlar ta'siriga barqaror emas. Shuning uchun agressiv va radiaktiv nurlar ta'sirlarga chidamli yangi ionitlarni sintez qilish dolzarb muammolardan biri hisoblanadi[11-14].

Ikkilamchi mahsulotlardan foydalanib import superplastifikator o'rnini bosuvchi mahsulot chiziqli oligomer polimetilennaftalin sulfonat natriy olish sharoitlari va uni beton aralashmalari mustahkamligiga ta'sirini o'rganish. Fazoviy tuzilishli polimetilennaftalin sulfokislotani esa sulfokationit sifatidagi xossalarini o'rganish muhim ahamiyatga ega.

Tadqiqot usullari

Xom-ashyo sifatida "Uz-KorGas Chemical" MChJ ga qarashli Ustyurt gaz-kimyo majmuasi ikkilamchi maxsuloti piroliz moyidan 210-230 ^oC oralig'ida olingan naftalin fraksiyasi qo'llanildi. Naftalin fraksiyasini sulfolash uchun laboratoriya ustanovkasi va polikondentsatsiyalash uchun germetik bosim ostida ishlaydigan uskunadan foydalanildi.

Olingan natijalarni tadqiq qilish uchun fizik-kimyoviy analiz usullardan foydalanildi.

Superplastifikator qo'llanilgan beton aralashmalari mustahkamligi GOST 10180-2012 usuli bo'yicha Gidravlik pres (MIG.1000.06 RU) pribori orqali aniqlandi.

Namunalarning IQ spektorlari IRTracer-100 spektrometrida 400-4000 sm^{-1} oraliq sohali olmosli/ ZnSe MIRacle 10 prizma bilan foydalanib o'lchandi.

Skanerlovchi elektron mikroskop (SEM) EVO MA-10 skanerlash elektron mikroskopida (Carl Zeiss, Germaniya) energiya dispersiyali rentgen (EDX) mikroanalizi (Oxford Instruments, Buyuk Britaniya) uchun mikroanalitik tizim bilan jihozlangan.

Kationitlarning termogravimetrik (TGA) va differentsial termik analizi (DTA) TG 209 F1 termogravimetrik analizatorida o'rganildi.

Sulfokationitni sinovga tayyorlash, solishtirma massasi, solishtirma hajmi, namligi, COE va DOE larini aniqlash GOST talablariga muovofiq aniqlandi [15-19].

Natijalar va muhokamasi

Polimetilennaftalin sulfokislotani sintez qilish uchun dastlabki homashyo sifatida uglevodorodlar pirolizi ikkilamchi mahsuloti- piroliz moyini fraksion haydash natijasida 210-230 ^oC da olingan naftalin fraksiyasidan foydalanildi.

Superplastifikatorni sintezlash uchun quyidagi jarayonlar amalga oshiriladi:

Piroliz moyini fraksion haydash orqali 210-230 ^oC oralig'idagi fraksiyalardan naftalin ajratib olindi va tozalandi.

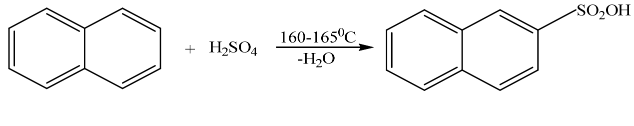
Olingan naftalin(tozalik darajasi 96%) 160-165 ^oC da 6 soat konsentrlangan sulfat kislota yordamida (mol nisbat 1:1,08) sulfirlandi, natijada to'q qora rangli sulfomassa olindi (1-reaksiya) [20].

Sulfomassa bosim ostida ishlaydigan idishga solinib distillangan suv bilan suyultirildi va 38% li formalin bilan(dastlabki naftalin va formaldegid mol nisbati 1;0,7) xarorat 110-120 ^oC da polikondensatlandi (2-reaksiya).

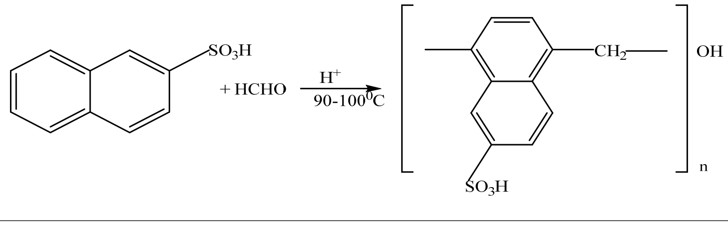
Chiziqli tuzilishli polimetilennaftalin sulfokislota oligomeri kaustik soda yordamida kuchsiz ishqoriy muhitgacha(pH=8) neytrallandi. (3-reaksiya).

Yuqorida keltirilgan jarayonlarning reaksiya tenglamalari quyidagicha:

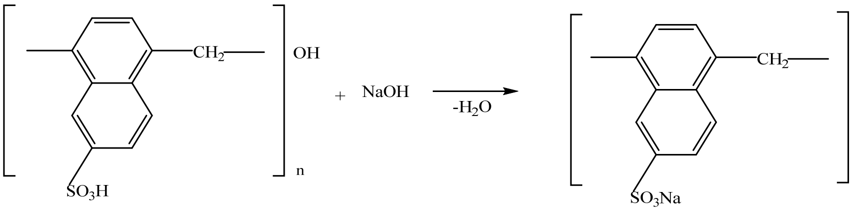
1-reaksiya:



2-reaksiya:



3-reaksiya:

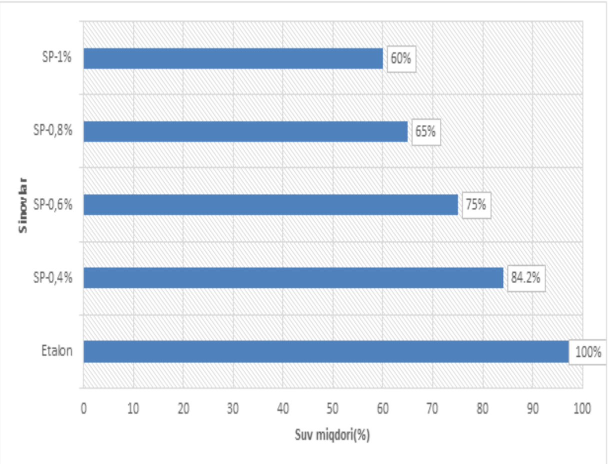


1-Jadval

Beton qorishmalarining tarkibi

№	Sement, g	Qum, g	Chaqiq tosh, g	Suv, g	SP sementga nisbatan, %	Konus cho'kmasi, sm
1.	2660	8750	3990	1115	-	18
2.	2660	8750	3990	935	0,4	18
3.	2660	8750	3990	845	0,6	18
4.	2660	8750	3990	725	0,8	18
5	2660	8750	3990	669	1	18

Olingan superplastifikatorning betonga ta'sirini o'rganish uchun quyidagi 5 ta tarkibli beton qorishma tayyorlandi (1-jadval). 1-qorishma etalon uchun, 2-qorishma 0,4% superplastifikator, 3-qorishma 0,6% superplastifikator, 4-qorishma 0,8% superplastifikator, 5-qorishmaga 1% superplastifikator sement miqdoriga nisbatan qo'shilgan holda sinaldi. Sinovda beton qorishmasining konus cho'kmasini П4 ya'ni 18 smda ushlagan holda suv sarfini kamaytirish va mustahkamligini oshirish natijalari o'rganildi.



1-rasm. Beton qorishmasi uchun sarflangan suv miqdori, %.

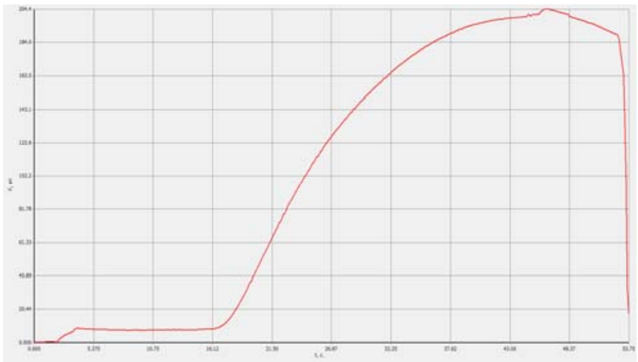
Etalon uchun olingan betonning konus cho'kmasi 18 sm pasayishi uchun sarflangan suv miqdoriga nisbatan 0,4% SP qo'shilganda 16,2% ga, 0,6% SP qo'shilganda 25% ga, 0,8% qo'shilganda 35% ga, 1% qo'shilganda 40% gacha kamaytirdi.

Yuqorida o'tkazilgan sinovlar natijasida olingan beton qorishmalari 10x10x10 sm o'lchamdagi qoliplarga quyildi. Olingan namunalar 28 kundan so'ng mustahkamligi GOST 10180-2012 usuli bo'yicha Gidravlik pres pribori orqali aniqlandi [21] (2-jadval). Namunalarning mustahkamligi 2-6-rasmlarda keltirilgan.

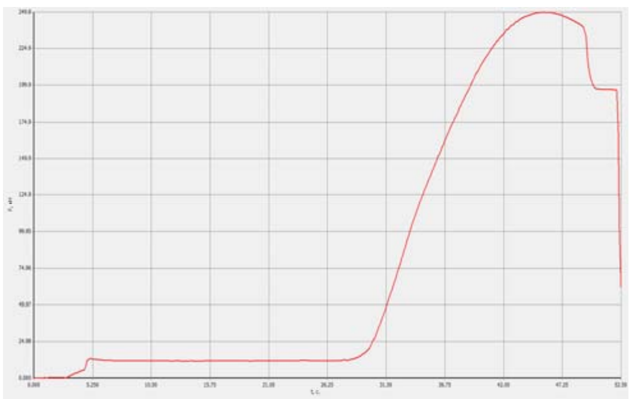
2-jadval

GOST 10180-2012 bo'yicha 28 kunlik namunalarning mustahkamligi

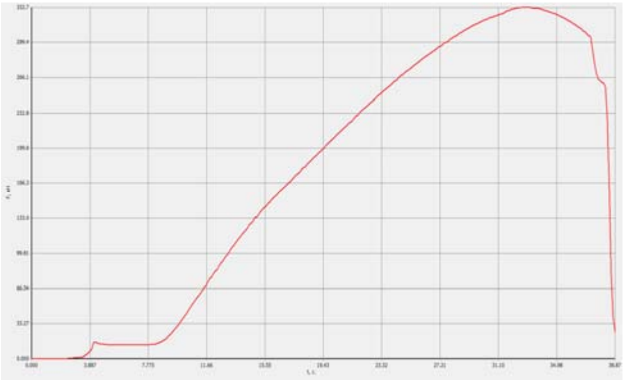
Namuna raqami	Maksimal kuch, $P_{мах}$, кН	Siqilish kuchi, $R_{сж}$, Мпа	Siqilish moduli, E_c, МПа
1(Etalon)	204.466	20.447	328.483
2(SP-0,4%)	249.896	24.990	355.144
3(SP-0,6%)	332.700	33.270	396.561
4(SP-0,8%)	377.022	37.702	376.516
5(SP-1 %)	274.206	27.421	533.264



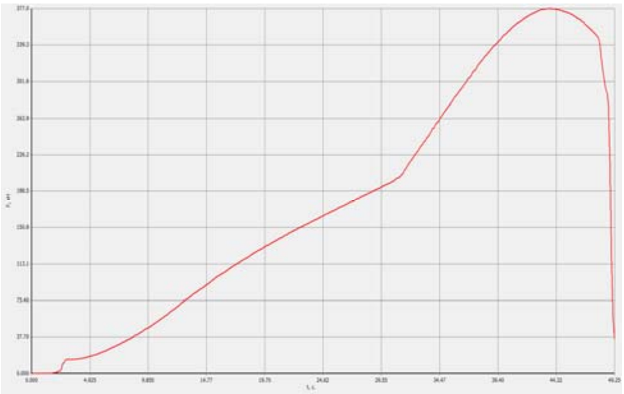
2-rasm. 10x10x10x см ўлчамли эталон намуналарнинг узоқ муддат қотишига боғликлиги.



3-rasm. 10x10x10 см ўлчамли намуналарнинг 0,4% SP узоқ муддат қўшилганда қотишига таъсири.



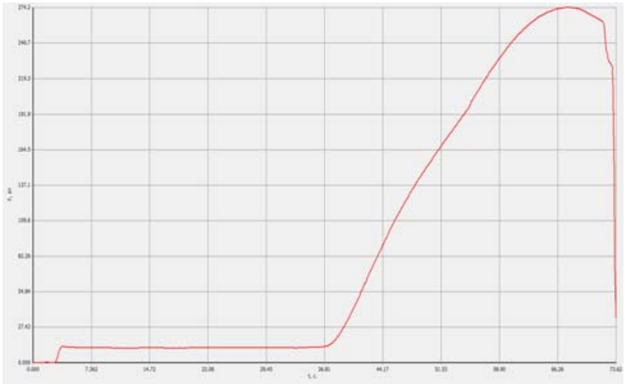
4-rasm. 10x10x10 см ўлчамли намуналарнинг 0,6% SP узоқ муддат қўшилганда қотишига таъсири.



5-rasm. 10x10x10 см ўлчамли намуналарнинг 0,8% SP узоқ муддат қўшилганда қотишига таъсири.

Olingan natijalar shuni ko'rsatadiki, piroliz sanoati ikkilamchi mahsuloti asosida olingan superplastifikator betonning 28 kundan keying mustahkamligini etalonga nisbatan 0,4% qo'shilganda 22,2% ga, 0,6% qo'shilganda 62,7% ga, 0,8% qo'shilganda 84,39 % ga, 1 % qo'shilganda 34,4 % ga oshirishi aniqlandi. Olingan natijalardan xulosa qilib eng optimal tarkib sifatida beton tarkibiga 0.8% SP qo'shilgan holat deb topildi.

Sintez qilingan superplastifikatorning IQ spektri olindi va tahlil qilindi (7-расм).



6-rasm. 10x10x10 см ўлчамли намуналарнинг 1% SP узоқ муддат қўшилганда қотишига таъсири.

Yuqoridagi IQ spektrning tahlili shuni ko'rsatadiki: 3428,5 sm^{-1} soxada –OH guruhining valent tebranishi; 3069,74 sm^{-1} soxada aromatik yadrodagi C-H bog'ining valent tebranishi; 2924,59 sm^{-1} soxada –CH_2- guruhining assimetrik valent tebranishi; 1117,76 sm^{-1} soxada -SO_3Na guruhning valent tebranishini ko'rishimiz mumkin [22, 23].

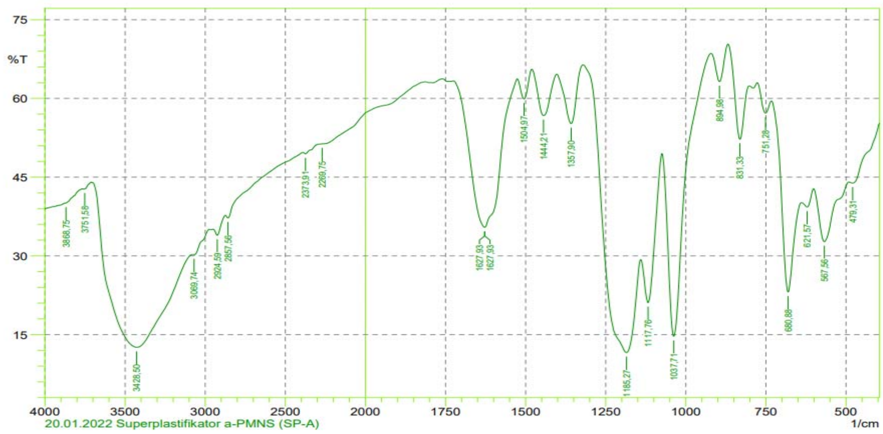
Sulfokationitni sintezlash uchun quyidagi jarayonlar amalga oshiriladi:

Piroliz moyini fraksion haydash orqali 210-230 ^oC oralig'idagi fraksiyalardan naftalin ajratib olindi va tozalandi.

Olingan naftalin(tozalik darajasi 96%) 160-165 ^oC da 8-10 soat konsentrlangan sulfat kislota yordamida (mol nisbat 1:2,5) sulfirlandi, natijada to'q qora rangli sulfomassa olindi. (1-reaksiya)

Sulfomassa bosim ostida ishlaydigan idishga solinib, 38% li formalin bilan(dastlabki naftalin va formaldegid mol nisbati 1;2) xarorat 110-120 ^oC, bosim 20-40ATM da polikondensatlandi. (2-reaksiya).

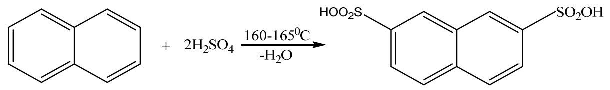
Suvda erimaydigan qattiq polikondensat mexanik maydalanib polikondensatsiyani oxiriga yetkazish uchun 12 soat 90-95 ^oC da qizdirildi. (3-reaksiya).



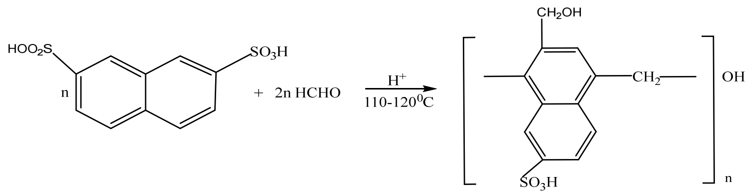
7-rasm. Chiziqli polimetilennaftalin sulfonat natriyning IQ spektri.

Yuqorida keltirilgan jarayonlarning reaksiya tenglamalari quyidagicha:

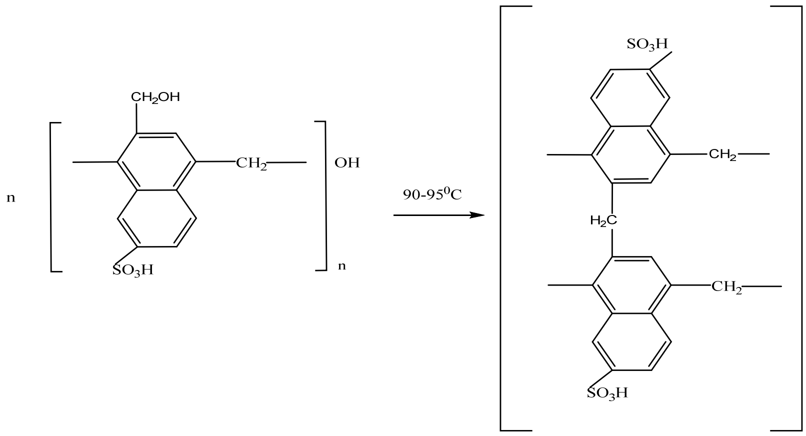
1-reaksiya:



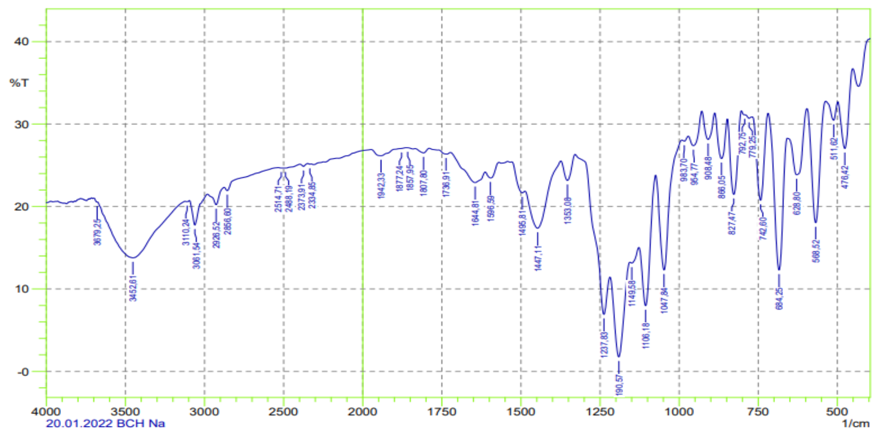
2-reaksiya:



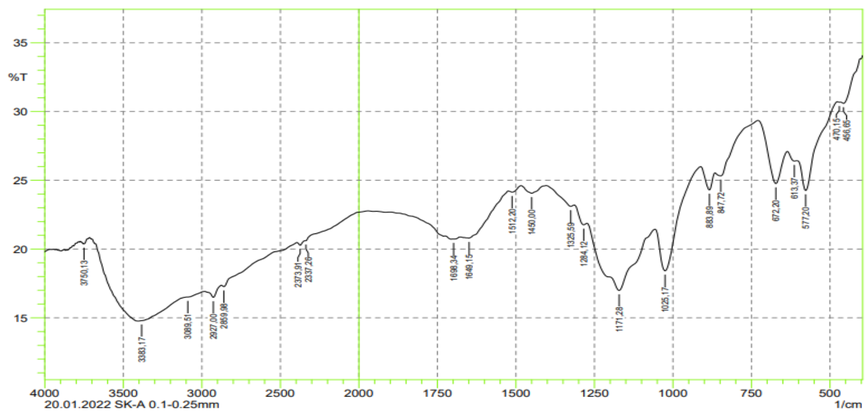
3-reaksiya:



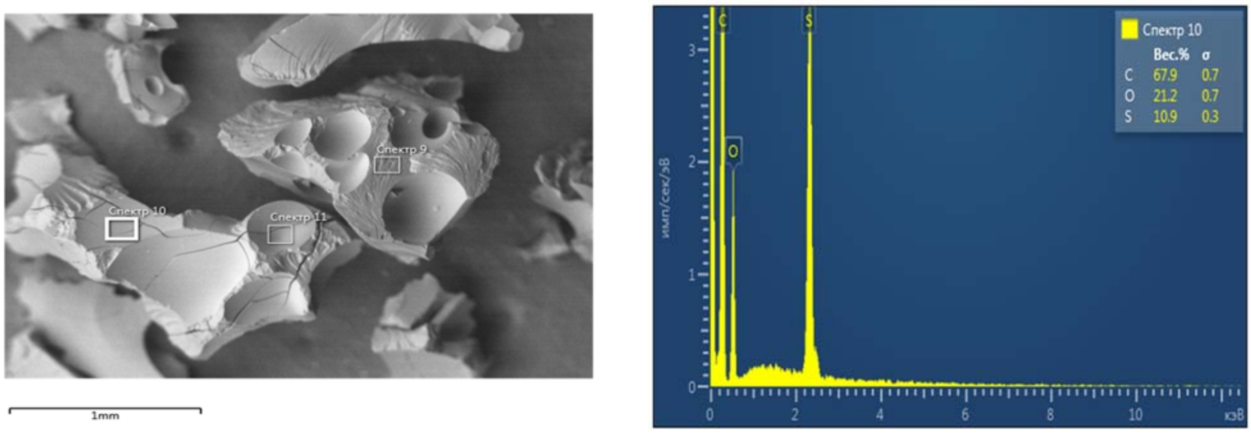
Olingan mahsulotlar tuzilishi IQ spektroskopiya yordamida tasdiqlandi.



8-rasm. 2,7-disulfonaftalinning IQ spektri.



9-rasm. Fazoviy polimetilennaftalin sulfokislotaning IQ spektr tahlili.



10-rasm. Sulfokationitning sirt tuzilishi va element tarkibi.

IQ spektrda 3061,54 sm^{-1} soxada aromatik yadrodagi C-H bog'ining valent tebranishi, 1106,18 sm^{-1} soxada $-SO_3H$ guruhning valent tebranishi kuzatilgan.

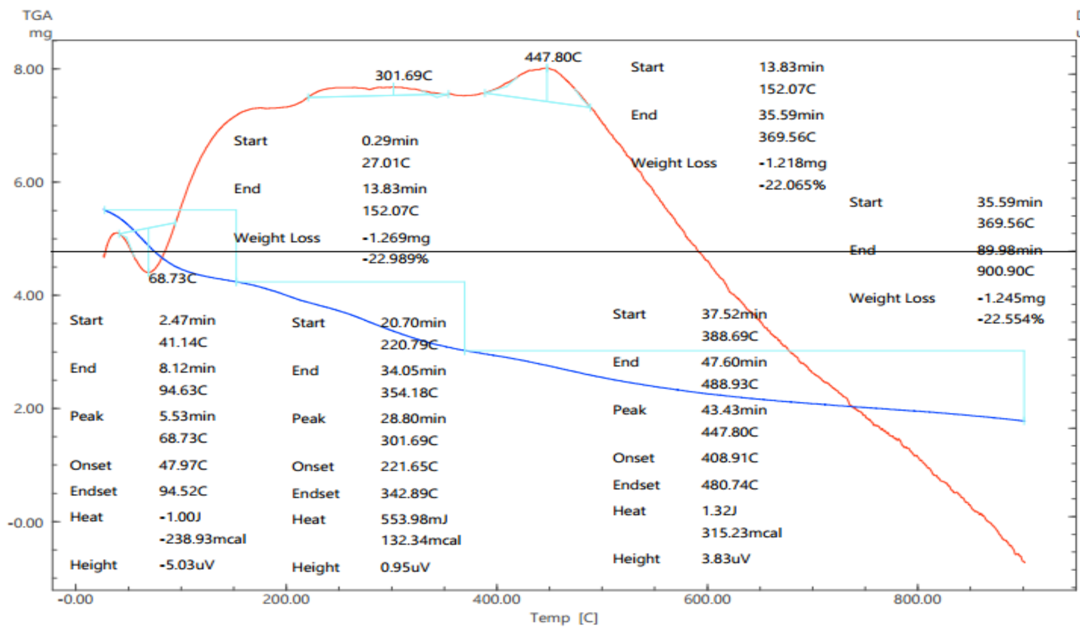
Olingan IQ spektri ma'lumotlaridan shunday xulosa qilish mumkinki, reaksiya davomida guruhlarning kirib kelishi shu gurhlarga xos valent va deformatsion tebranish hosil qilganini ko'rishimiz mumkin.

Sintez qilingan sulfokationitning morfologiyasi va sirt tuzilishi va element tarkibi SEM (skanerlovchi elektron mikroskopi) yordamida aniqlandi.

Sulfokationitining sirt SEM tahlilining natijalari shuni ko'rsatadiki, sulfokationit tarkibida mezog'ovaklarning mavjudligini ko'rish mumkin. Sulfokationitning element tahlili uning tarkibida 69,9% C, 21,2% O va 10,9% S borligi aniqlandi.

Sulfokationitning termik barqarorligi termogravimetrik usul bilan o'rganildi.

Rasmda keltirilgan ma'lumotlar uch bosqichda vazn yo'qotish bilan namuna tuzilishining o'zgarishini ko'rsatadi, birinchisi 27,01-152,07 ^oC, bu diapazonda 22,989% gacha, ikkinchi bosqich esa 152,07-369,56 ^oC, bu oraliqda 22,065%, 369,56-900,9 ^oC gacha esa 22,554% modda massasini yo'qotdi. Olingan sulfokationit 900 ^oC gacha qizdirilganda umumiy 67,608% massasini yo'qotishi aniqlandi. O'rganilayotgan kationitlarning differentsial termik egrisi ikki endotermik tepalik va ikkita ekzotermik tepalik bilan ifodaladi. Birinchi endotermik ta'sir 41,14-94,52 ^oC da sodir bo'lgan bu kationitdan gigroskopik va kristalizatsion suvning yo'qolishi bilan izohlanadi. Ikkinchi endotermik tepalik 600 ^oC dan yuqori haroratda paydo bo'ladi, bu ionitning destruksiyasi bilan tushintirish mumkin. Ikkita ekzotermik tepaliklar 220,79-354,14 ^oC va 388,69-488,93 ^oC larda kuzatilib bu oraliqlarda kationitdagi faol furuhlar –OH, $-SO_2OH$ guruhlarining o'zaro poli-



11-rasm. Sulfokationitning termogrammasi.

3-Jadval

Sintez qilingan sulfokationit va KU-2-8(import) sulfokationitlarning ekspluatatsion xossalari

Kationit turi	Solishtirma massasi, g/dm^3	Namligi, %	Solishtirma hajmi, sm^3/g	Umumiy statik al-mashinish sig'imi, mg-ekv/g	Dinamik al-mashinish sig'imi, mol/m^3
	O'rganish usuli				
	GOST 10898.2-74	vlagamer XY-100MW	GOST 10898.4-84	GOST 20255.1-89	GOST 20255.2-89
ASO	650-720	62,5	4,8	4,6	475-490
KU-2-8 (nazorat)	750 – 800	48-58	2,8	4,6-4,8	500-520

kondensatlanishi natijasida suvning ajralib chiqishi bilab bog'liq. KU-2 kationit uchun 353–413 K da energiya yutilishi bilan endotermik tepalik kuzatiladi va uning destruksiyasi esa 423 K da kuzatiladi [24-26]. Shunday qilib, naftalin asosidagi olingan kationitning termal barqarorligi KU-2 kationitdan yuqori ekanligini ko'rish mumkin.

Sulfokationitning quyidagi ekspluatatsion xossalari o'rganildi:

kationitning solishtirma massasi
kationitning solishtirma hajmi
kationitning namligi
kationitning statik almashinish sig'imi
kationitning dinamik almashinish sig'imi

Olingan katonitlarni sinovga tayyorlash uchun GOST 10896-78 xalqaro standart bo'yicha ishlar amalga oshirildi. Solishtirish maqsadida KU -2-8 sulfokationiti olindi (3-jadval).

Jadvaldan ko'rinadiki, sintez qilingan ASO sulfokationitlarning asosiy ekspluatatsion xossalari import sulfokationit KU-2-8 ning statik va dinamik almashinish qobilyatiga yaqinligini ko'rishimiz mumki.

Xulosa

Uglevodorodlar piroliz jarayoni ikkilamchi maxsuloti piroliz moyidan oqilona foydalanish maqbul usuli polimetilennaftalin sulfokislota ishlab chiqarish va bu polimerning chiziqli oligomerini superplastifikator sifatida betonda, fazoviy polimeri sulfokationit sifatida foydalanish mumkinligi aniqlandi.

Etalon uchun olingan betonning konus cho'kmasi 18 sm pasayishi uchun sarflangan suv miqdoriga nisbatan 0,4% SP qo'shilganda 16,2% ga, 0,6% SP qo'shilganda 25% ga, 0,8% qo'shilganda 35% ga, 1% qo'shilganda 40% gacha kamaytirishi aniqlandi.

Sintez qilingan superplastifikator betonning 28 kundan keying mustahkamligini etalonga nisbatan 0,4% qo'shilganda 22,2% ga, 0,6% qo'shilganda 62,7% ga, 0,8% qo'shilganda 84,39% ga, 1% qo'shilganda 34,4% ga oshirishi aniqlandi. Olingan natijalardan xulosa qilib eng optimal tarkib sifatida beton tarkibiga 0.8% SP qo'shilgan holat deb topildi.

Kationitlarning muhim ekspluatatsion xossasi COE = 4,6 mg-ekv/g, DOE = 475-490 mol/m^3.

Polimetilennaftalin sulfokislota tarkibidagi funksional guruhining mavjudligi IQ spektori yordamida isbotlangan va sulfokationit SEM yordamida morfologiyasi o'rganildi.

Sulfokationitning termik barqarorligi termogravimetrik usul bilan o'rganildi. Naftalin asosidagi olingan kationitning termal barqarorligi KU-2 kationitdan yuqori ekanligi aniqlandi.

Sintez qilingan sulfokationitlarning statik va dinamik almashinish sig'imi KU-2-8 sulfokationiti xossasiga yaqinligi o'rganildi.

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