

تحضير البولي أكريل أمايد و بعض بوليمراته المشتركة الحاوية على الزئبق و دراسة تطبيقاتها الصناعية

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الخلاصة:-

-فيनाيل كاربازول Nتم تحضير البولي اكريل امايد و بعض بوليمراته المشتركة مع الستايرين و بواسطة البلمرة الجذرية ، كما تم اجراء بعض التحويلات الكيميائية باستخدام البروم و الزانثيدرول و التفاعل مع الزئبق باستخدام أوكسيد الزئبق و خلاص الزئبق على البولي اكريل امايد و بوليمراته المشتركة.

أجريت هذه الدراسة لإزالة ثنائي بنزو ثايوفين من نموذج محضر للنفط الخفيف (البنزين المحتوي على DBT، 3080 جزء في المليون كبريت) باستخدام البوليمرات الحاوية على الزئبق.

أستخدم جهاز الامتصاص الذري لمعرفة تركيز الكبريت في محاليل ثنائي بنزو ثايوفين بعد المعاملة مع البوليمرات الحاوية على الزئبق. كما تم تشخيص البوليمرات المحضرة باستخدام طيف الاشعة تحت الحمراء.

الكلمات الدالة:- بولي أكريل أمايد ، بوليمرات مشتركة ، بوليمرات حاوية على الزئبق ، إزالة مركبات الكبريت.



Preparation of Some mercurated Polyacrylamide and It's Copolymers and Their Industrial Applications

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Abstract:-

polyacrylamide and some of its copolymers with styrene and N-vinyl carbazole have been prepared by free radical polymerization and some chemical modifications by using bromine, xanthhydrol and mercuration with mercury oxide and mercuric acetate have been done on polyacrylamide and its copolymers.

This study was carried out to remove dibenzothiophene from model light oil (benzene containing DBT, 3080 ppm. sulfur content) by using the mercurated polymers. The analysis of the resulting treated DBT solution to know the sulfur concentration was obtained by using atomic absorption





The mercurated polymers were identified spectroscopically using infrared spectroscopy.

Keywords: polyacrylamide, copolymers, mercurated polymers, esulfurization.

1-Introduction:-

Chemical modification of polymers is one of the methods used to produce new polymeric materials to extend application of the existing polymers does not have the required properties, and there is difficulty in synthesizing a new monomer and/or the monomer is not stable under the polymerization conditions. The applications of the product include non-material uses such as polymeric reagents which consist of a reactive function bound to an insoluble support, functionalized resins which are used as an exchangers, chelating agents, reagent for organic synthesis, catalysts, and vehicles for carrying active molecules and drugs.^[1]

More particularly, the invention relates to a new class of homo and copolymers which contain mercuric ions aromatically bound therein. The products produced in accordance with this invention find utility as carries in biopolymer applications, i.e. as carrier gels for removing sulfur-containing groups from mixtures, in tracing studies, as carrier for biologically active substances and the like.^[2]





Crude oil is the largest and most widely used source of energy in the world. However, such crudes contain sulfur, typically in the form of organic sulfur compounds. The sulfur content and the API gravity are two properties which have a great influence on the value of the crude oil; the sulfur content is expressed as a percentage of sulfur by weight and varies from less than 0.1% to greater than 5% depending on the type and source of crude oils.^[3]

The most abundant heterocyclic sulfur compounds identified in petroleum includes sulfides, thiophenes, benzothiophenes and naphthobenzothiophenes.

During the partial or complete combustion of a sulfur-containing residual oil, the sulfur gets converted to SO_2 and SO_3 (typically referred to as SO_x). During the combustion or pyrolysis process, the sulfur emissions from a typical high sulfur residual oil can be extremely high; additional processes such as flue gas desulfurization are needed to clean up these emissions and bring the SO_x content of effluent gases to acceptable levels. This requires an additional investment of capital, as well as additional operating expense, to continue operating industrial units that use the sulfur-containing residual oil^[4]

One of the easily and fast method to remove sulfur from diesel oil is the adsorption desulfurization process.^[5] It is one of the methods which can be used to achieve ultra-low sulfur content in diesel fuel. It is





considered the most effective method for the removal of the trace amounts of residual sulfur in fuels after hydro desulfurization treatment.^[6]

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Chemical modification of polymers is one of the methods used to synthesize new polymeric compounds. It is the most active fields of research in polymer sciences for the past 30 years and enables us to introduce functional or reactive groups into polymers, to alter polymer surfaces, to provide side chain substituents, etc,. Important products of polymer modification include polymeric reagents, functionalised resins, catalyst and media for trapping reaction intermediates organometallic polymers acid carrier for active molecules.^[7]

The mercuration of polymers is a suitable method for the preparation of selective adsorbents.^[8]

2-Experimental:-

2-1 Materials and instruments:-

All the chemicals used have been supplied by Fluka and Aldrich.

Infrared spectra have been recorded by shemadzue FT-IR-8400 s Fourier transform Infrated spectrophotometer in the range between (4000-250 cm⁻¹).





Varian-AA-240FS Fast Sequential Atomic Absorption Spectroscopy, Australian, used for measuring the concentration of mercury and sulfur ions in solutions in N.G.C / Kirkuk-Iraq.

All the samples have been dried at 50-65 °C by using drying oven (Memmert).

2-2-Preparation of Polyacrylamide(A-1):-

(4.00 g.) of the monomer (acrylamide) was dissolved in a mixture of toluene (30 ml) and methanol (10 ml) in a round bottom flask and then added to the reaction mixture (0.02 g.) of the initiator (dibenzoyl peroxide). The reaction mixture was refluxed in a water bath at (75 °C) with constant stirring for 30 minutes. The polyacrylamide was filtered and washed well with a mixture of toluene and methanol several times. The weight of product after drying was (2.98 g).

2-3-Mercuration of polyacrylamide (A-2):-

(0.0047 mole, 0.34 g.) of polyacrylamide was dissolved in (20 ml) of distilled water in a round bottom flask, then (0.0015 mole, 0.2535 g.) of mercury oxide (II) was added to the resulting mixture. The reaction mixture was refluxed in a water bath at (75 °C) with constant stirring for 7 hrs. The resulting mixture was filtered after the disappearance of orange color of the mercury oxide and washed well with warm water several times and with very dilute solution of acetic acid to get rid of the remaining unreacted mercury oxide. The filtrate was examined for mercury with a dilute solution of sodium hydroxide to make sure that



there is no mercury ions in it. The weight of product after drying was (0.48 g.).

2-4-Preparation of N-bromopolyacrylamide (A-3):-

(0.10 g.) of polyacrylamide was dissolved in (10 ml) of distilled water in a round bottom flask then (0.3 ml) of bromine was added to the solution with stirring at room temperature for (6 hrs.) where it was noted the disappearance of bromine color. The polymeric product was filtered and washed well with distilled water several times. The weight of product after drying was (0.08 g.).

2-5-Mercuration of N-bromopolyacrylamide (A-4):-

(0.0002 mole, 0.099 g.) of N-bromopolyacrylamide was added in a round bottom flask and then added (5 ml.) of distilled water and (0.0001 mole, 0.0216 g.) of mercury oxide (I). The reaction mixture was refluxed in a water bath at (75 C°) with constant stirring for (7 hrs). The resulting precipitate was filtered after the disappearance of the orange color of the mercury oxide and washed with warm water several times and with very dilute solution of acetic acid to get rid of the remaining unreacted mercury oxide. The filtrate was examined with a dilute solution of sodium hydroxide to make sure the disappearance ions. The weight of product after drying was (0.0580 g.).

2-6-Preparation of Polyxanthylacrylamide (A-5):-

(0.25 g.) of Xanthhydrol was dissolved in (3.5 ml.) of glacial acetic acid; an oil was separated (as is sometimes the case with commercial material), allowed to settle for a short time and the supernatant solution was decanted, then (0.25 g.) of polyacrylamide was added, shaken and allowed to stand. The reaction mixture was refluxed in a water bath at (60 C^o) with constant stirring for (2 hrs.).

A viscous liquid was obtained, after cooling a little methanol was added, the resulting mixture was filtered and the precipitate was washed off with distilled water several times and with a little methanol. The weight of product after drying was (0.3291 g.).

2-7-Mercuration of Polyxanthylacrylamide Using Mercury acetate in THF (A-6):-

(0.001 mole, 0.2533 g.) of polyxanthylacrylamide were added to (10 ml.) of THF in a (250 ml.) flask with stirring for (30 min.) at room temperature. Then, (0.001 mole, 0.3186 g.) of mercury acetate and (0.5 ml.) acetic acid were added to the reaction mixture. The content was refluxed for (24 hrs.) and the resulting mixture poured into a (75 ml.) of water. The polymer was collected and washed with warm water several times and with very dilute solution of acetic acid to get rid of the remaining unreacted mercury oxide. The filtrate was examined for mercury with a dilute solution of sodium hydroxide to make sure that there is no mercury ions in it. The weight of product after drying was (0.3240 g.).

2-8-Preparation of Poly(acrylamide-co-styrene) (A-7):-

(0.50 g.) of acrylamide and (0.50 g.) was dissolved in (3 ml.) of DMF in a round bottom flask and then (0.01 g.) of the initiator (dibenzoyl peroxide) was added. The reaction mixture was refluxed in a water bath at (75 C^o) with constant stirring for (3 hrs.). The reaction mixture was added to (150 ml.) of cold water, the copolymer was filtered and dried. The weight of product after drying was (0.5564 g.).

2-9-Mercuration of Poly(acrylamide-co-styrene) Using Mercury acetate in THF (A-8):-

(0.52 g.) of copolymer was dissolved in (10 ml.) of THF in a round bottom flask and then added to the solution (1.5930 g.) of mercury acetate and (1 ml.) of acetic acid. The reaction mixture was refluxed in a water bath at (70 C^o) with constant stirring for (24 hrs). The resulting precipitate was added to (100 ml.) of distilled water and filtered after and had been washed off with warm water several times and with very dilute solution of acetic acid to get rid of the remaining mercury oxide. The filtrate was examined with a dilute solution of sodium hydroxide to make sure that there is no mercury ions in it. The weight of product after drying was (0.6130 g.).

2-10-Copolymerization of Acrylamide and N-vinylcarbazole (A-9):-

(1.50 g.) of acrylamide and (1.50 g.) of N-vinylcarbazol were dissolved in a mixture of toluene (23 ml) and methanol (7 ml) in a round bottom flask and then added to the reaction mixture (0.0150 g.) of the

initiator (dibenzoyl peroxide). The reaction mixture was refluxed in a water bath at (75 C^o) with constant stirring for 30 minutes. The copolymer was filtered and washed well with a mixture of toluene and methanol several times. The weight of product after drying was (1.4509 g).

2-11-Mercuration of Poly(acrylamide-co-N-vinylcarbazole) (A-10):-

(1.70 g.) of poly(acrylamide-co-N-vinylcarbazol) were added to (10 ml.) of Dioxan in a (250 ml.) flask with stirring for (30 min.) at room temperature. Then, (1.267 g.) of mercury oxide and (8 ml.) acetic acid were added to the reaction mixture. The content was refluxed for (24 hrs.) and the resulting mixture was poured into a (200 ml.) of water. The mercurated polymer was collected and washed with warm water several times and with very dilutes solution of acetic acid to get rid of the remaining unreacted mercury oxide. The filtrate was examined for mercury with a dilute solution of sodium hydroxide to make sure that there is no mercury ions in it. The weight of product after drying was (1.901 g.).

2-12-Desulfurization (Using Different Conditions):-

Time:- (10 ml.) Solution of (Dibenzothiophene in Benzene) (3083 ppm) was mixed with (0.1 g.) of polymer (All mercurated polymers was used for desulfurization) in a round bottomed flask with stirring for (4,8,12,16,20,24 hrs.) at room temperature. The resulting solution was filtered to separate the polymer from filtrate. The filterate was analyzed

for sulfur content. This step is to choose the proper contact time that gives highest sulfur removal.

Temperature:- (10 ml.) solution of (Dibenzothiophene in Benzene) was mixed with (0.1 g.) of polymer (All mercurated polymers was used for desulfurization) in a round bottomed flask with stirring for (24 hrs.) with raise the temperature to (35 C^o) and (50 C^o). The resulting solution was filtered to separate the polymer from filtrate. The filtrate was analyzed for sulfur content. This step is to choose the proper temperature that gives highest sulfur removal.

The Amount Of Absorbent:- (10 ml.) solution of (Dibenzothiophene in Benzene) was mixed with (0.1,0.3,0.5 g.) of polymer (All mercurated polymers was used for desulfurization) in a round bottomed flask with stirring for (24 hrs.) at (50 C^o). The resulting solution was filtered to separate the polymer from filtrate. The filtrate was analyzed for sulfur content. This step is to choose the proper amount of absorbent that gives highest sulfur removal.

3-Results an Discussion:-

3-1-Preparation of Polyacrylamide(A-1):-

In this study linear polyacrylamide was prepared by free radical polymerization of acrylamide using dibenzoyl peroxide as a radical initiator.

In the infrared spectrum of polyacrylamide, the two strong intensity bands appearing around 3386 cm^{-1} and 3199 cm^{-1} are undoubtedly associated with the N-H stretching vibrations. The corresponding modes in acrylamide are found at 3352 cm^{-1} and 3180 cm^{-1} .

The methylene group vibration useful to monitor the extend of polymerization. The calculated values 2933 cm^{-1} and 2864 cm^{-1} are assigned to asymmetric and symmetric CH_2 stretching vibrations, respectively.

In acrylamide (the monomer), these modes are observed at 3105 cm^{-1} and 3030 cm^{-1} . The lowering of the above frequencies in polyacrylamide compared to acrylamide is mainly due to the unsaturated linkage in monomer acrylamide. The weak intensity infrared band observed at 2786 cm^{-1} in polyacrylamide has been assigned to CH stretching mode.

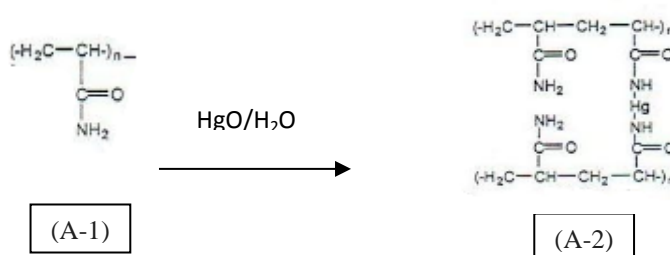
3-2-Mercuration of polyacrylamide (A-2):-

The amide nitrogen forms a covalent bond with mercury (II) ions but not with other metal ions. Generally, the amide nitrogen atom lacks sufficient electron donating character due to the adjacent electron withdrawing carbonyl group and it cannot normally form a coordination bond with transition metal ions under regular conditions.^[9]

The IR spectrum of free polyacrylamide and the mercured polyacrylamide can be compared with each other to check spectral

changes. An important spectral change observed between the spectra of free polyacrylamide and the mercurated polyacrylamide investigated here is the shifting of a strong broad band observed at 1672 cm^{-1} in free polyacrylamide to higher wave numbers and it's splitting into two bands at 1681 cm^{-1} , near 1652 cm^{-1} .

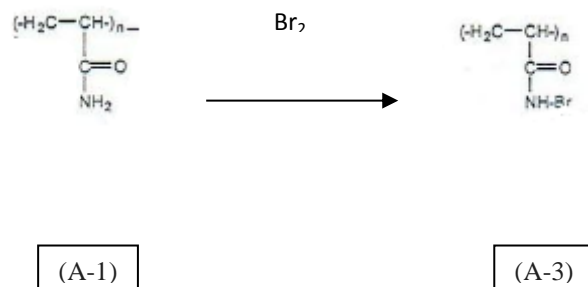
It was observed an increase in the weight of product, which indicates the entry of mercury in the polymer structure. Percent mercury was equal to (51.63%).



3-3-Preparation of N-bromopolyacrylamide (A-3):-

The IR spectrum of poly(N-bromo acrylamide) showed those characterization peaks of polyacrylamide with the intensity of the broad band in the region $3100\text{-}3500\text{ cm}^{-1}$ decreasing considerably.

This polymeric reagent was obtained as a yellowish-white powder and was stable and did not lose bromine under laboratory conditions and could be stored for a long time.



3-4-Mercuration of N-bromopolyacrylamide (A-4):-

Poly(N-bromo acrylamide) was also mercured by using of mercury oxide (II).

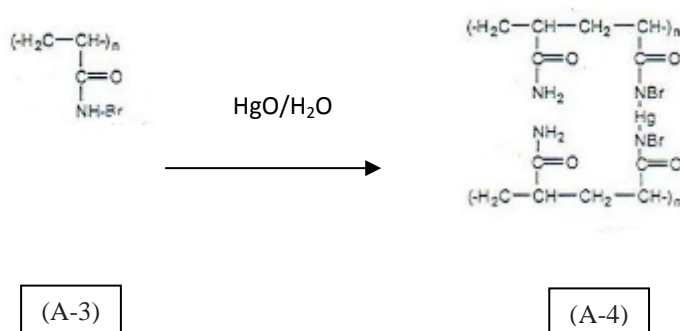
The IR spectrum of poly(N-bromo acrylamide) and the mercured poly(N-bromo acrylamide) can be compared with each other to check spectral changes.

The comparison of the IR spectrum of free poly(N-bromo acrylamide) and the mercured poly(N-bromo acrylamide) in the NH_2 vibration region near $3395\text{-}3197\text{ cm}^{-1}$ reveals a significant increase of the positions of $\nu_{\text{as}}(\text{NH}_2)$ and appreciable increase in $\nu_{\text{s}}(\text{NH}_2)$ vibrations to higher wave numbers accompanied by splitting into more sharp components.

An important spectral change observed between the spectra of free poly(N-bromo acrylamide) and the mercured poly(N-bromo acrylamide) investigated here is the changing of a bands observed at 1633 cm^{-1} and 1596 cm^{-1} in free poly(N-bromo acrylamide), it's appear more

sharp in 1629 cm^{-1} and 1596 cm^{-1} of the mercurated poly (N-bromoacrylamide).

It observed an increase in product weight, which indicates the entry of mercury in the polymer structure. Percent mercury was equal to (24.93%).

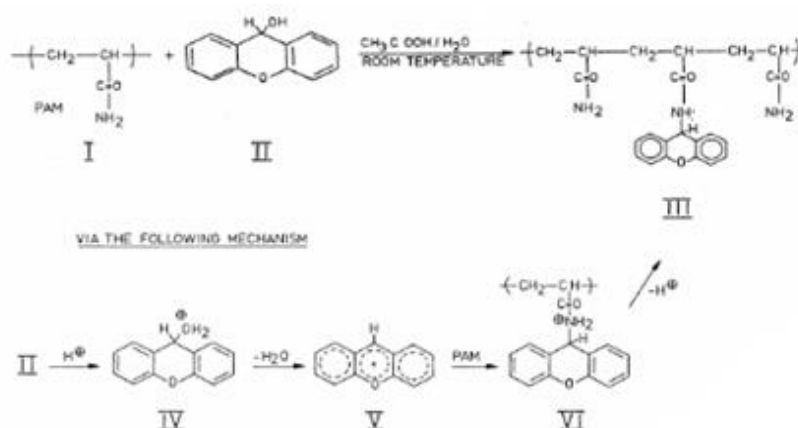


3-5-Preparation of Polyxanthylacrylamide (A-5):-

Amide group containing polymers such as polyacrylamide are modified, by reacting the polymer in aqueous solution with organic dye molecules which can form stable carbo-cations.

One of the most preferred dye molecules is xanthhdrol. (Scheme-1) depicts a mechanism for forming a labeled polymer from polyacrylamide and 9-xanthhydrol.^[10]

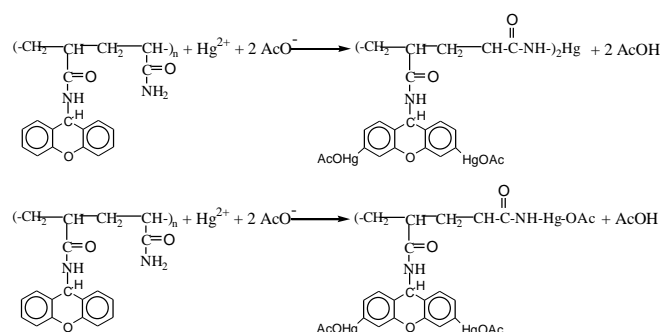
IR spectrum of the resultant copolymer showed absorption of aromatic C-C stretching in rings of xanthene group at 1454 cm^{-1} , C-O stretching vibration at 1336 cm^{-1} .



(Scheme-1):- Mechanism for forming polyxanthylacrylamide^[1,1]

3-6-Mercuration of Polyxanthylacrylamide Using Mercury acetate in THF (A-6):-

Apparently this reaction involves crosslinking by mercuric ions via the free amide groups as shown below:-



Due to the presence of phenyl groups of xanthene groups in the polymer backbone, the reaction taken place via electrophilic substitution in the phenyl group.

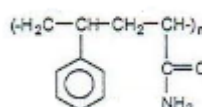
The IR of the mercurated polyxanthyl-acrylamide showed the presence of mercuric acetate group in the loaded copolymer confirmed by band observed at 1691 cm^{-1} can be assigned to the C=O stretching vibration of the acetate group. In contrast the C=O stretching vibration of the amide group appears at 1650 cm^{-1} .

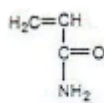
It was observed an increase in product weight, which indicates the entry of mercury in the polymer structure. Percent mercury was equal to (12.72%).

3-7-Preparation of Poly(acrylamide-co-styrene) (A-7):-

This copolymer was characterized through the infrared spectrum which is rather different from the spectrum of polystyrene without acrylamide modification.

In copolymer spectra, there are characteristic and intense peaks found at 3348 cm^{-1} , 1676 cm^{-1} and 1026 cm^{-1} indicating the N-H stretching , C=O stretching and C-N stretching due to acrylamide group.





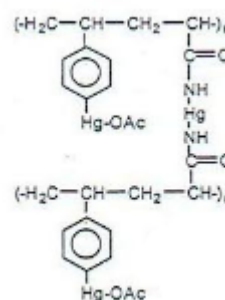
(A-7)

3-8-Mercuration of Poly(acrylamide-co-styrene) Using Mercury acetate in THF (A-8):-

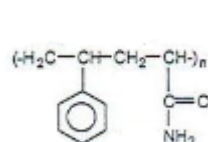
This reaction of poly(styrene-co-acrylamide) involves crosslinking as polyacrylamide and its copolymers by mercuric ions via the free amide groups. Due to the presence of styrene repeating units in the copolymer backbone, i.e. the presence of phenyl groups, the reaction takes place via electrophilic substitution in the phenyl group.

The presence of the mercuric acetate group in the loaded copolymer is confirmed by the FT-IR spectra, the band observed at 1670 cm^{-1} can be assigned to the C=O stretching vibration of the acetate group. Although the N-H plane bending vibration is also observed at the same frequency, after the mercury sorption this band becomes stronger. In contrast the C=O stretching vibration of the amide group appears at 1593 cm^{-1} . The peak in 827 cm^{-1} refer to 1,4-disubstituted benzene ring.

An increase in weight of product was observed, which indicates the



entry of mercury in the copolymer structure. Percent mercury was equal to (24.48%).

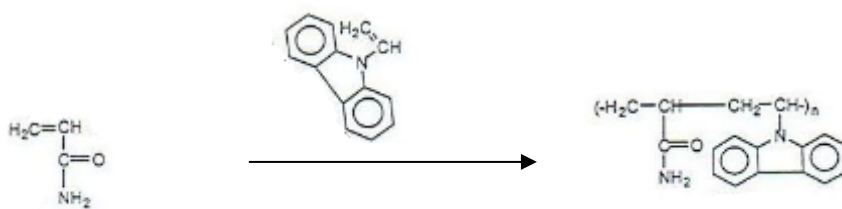


(A-7)

(A-8)

3-9-Copolymerization of Acrylamide and N-vinylcarbazole (A-9):-

In FT-IR spectrum of poly(acrylamide-co N-vinylcarbazole), there are shifts occurs in the vibrations as we can see from copolymer. The vibration for hydrogen bonded NH group in this copolymer has been approximately shifted to 3382 cm from 3386 cm⁻¹, whereas the band corresponding to C=O has been blue shifted to 1668 cm⁻¹ from 1672 cm⁻¹. The bending vibration band of NH has been blue shifted to 1330 cm⁻¹ from 1348 cm⁻¹.

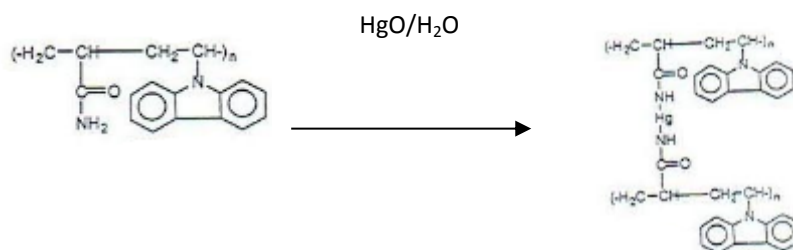


3-10-Mercuration of Poly(acrylamide-co-N-vinylcarbazole) (A-10):-

The mercured poly(acrylamide-co N-vinylcarbazole) was obtained by the reaction of this copolymer with mercuric oxide and forming a covalent bond between amide nitrogen and mercury (II) ions and the electrophilic attack by mercury acetate on benzene ring of carbazole.

The comparison of the IR spectrum of poly(acrylamide-co-N-vinylcarbazole) and the mercured copolymer in the NH_2 vibration region reveals a significant increase of the position to higher wave numbers, NH_2 stretching vibration at 3406 cm^{-1} , accompanied by splitting into more sharp components. An important spectral change observed in the spectra of poly (acrylamide-co-N-vinylcarbazole) and the mercured copolymer investigated here is the shifting of a bands observed at 1668 cm^{-1} and 1614 cm^{-1} in free copolymer to lower wave number at 1625 cm^{-1} , 1598 cm^{-1} in the mercured copolymer.

It was observed an increase in the weight of product, which indicates the entry of mercury in the polymer structure. Percent mercury was equal to (22.17%).





(A-9)

(A-10)

2-12-Desulfurization (Using Different Conditions):-

All mercurated polymers were used to desulfurize the solution of
(dibenzothiophene in benzene 3083 ppm).

Time:- All polymers gave the proper contact time that gives highest
sulfur removal at room temperature was (24 hrs.), as shown in table (3-
1):-

***Table (3-1):- Sulfur concentration in (dibenzothiophene solution) after
treated with mercurated polymers and Chelating polymers at different
Times***





Polymer	Time (hr.)	Sulfur (ppm)
A-2	4	2907.31
	8	2722.55
	12	2329.67
	16	1914.92
	20	1832.90
	24	1817.26
	4	2942.43
	8	2827.11
	12	2568.43
	16	2243.26
	20	2019.19
	24	2010.07
A-6	4	3002.44
	8	2737.20
	12	2516.59
	16	2294.29
	20	2147.64
	24	2147.12
A-8	4	2975.14
	8	2883.57
	12	2612.11
	16	2381.37
	20	2210.33
	24	2203.09
A-10	4	2919.12
	8	2776.10
	12	2501.11
	16	2185.86
	20	2082.09
	24	2066.04





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Temperature



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:- By raising the temperature of desulfurization from room temperature to (35 C⁰) and (50 C⁰).

It was observed a better desulfurization, as shown in table (3-2):-

Table (3-2):- Sulfur concentration in (dibenzothiophene solution) after treated with mercurated polymers and Chelating polymers at different Temperatures

Polymer	Temperature (C ⁰)	Sulfur (ppm)
A-2	25 (room temp.)	1817.26
	35	1604.97
	50	1552.44
A-4	25 (room temp.)	2010.07
	35	1883.77
	50	1769.82
A-6	25 (room temp.)	2147.12
	35	2011.08
	50	2004.21
A-8	25 (room temp.)	2203.09
	35	2151.37
	50	2103.71
A-10	25 (room temp.)	2066.04.
	35	1993.83
	50	1987.22

The amount of absorbent:- (0.1,0.3 and 0.5g.) of polymers were mixed with solution of (dibenzothiophene in benzene), for (24 hrs.), at (50 C°) with continuous stirring and then subjected to filtration process. The filtrate was analyzed for sulfur content and the results are shown in table (3-3):-

Table (3-3):- Sulfur concentration in (dibenzothiophene solution) after treated with mercurated polymers and Chelating polymers by using different amount of mercurated polymers

Polymer	Absorbent (g)	Sulfur (ppm)
A-2	0.1	1552.44
	0.3	1337.21
	0.5	1008.90
A-4	0.1	1769.82
	0.3	1434.34
	0.5	1130.86
A-6	0.1	2004.21
	0.3	1979.08
	0.5	1951.42
A-8	0.1	2103.71
	0.3	1898.22
	0.5	1783.65
A-10	0.1	1987.22



	0.3	1900.52
	0.5	1762.44

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