 Egypt. J. Chem. **Vol. 65,** No. 7, pp. 251 - 258 (2022)

Egyptian Journal of Chemistry
http://ejchem.journals.ekb.eg/

Improving the rheological properties of asphalt by cellulose acetate

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Abstract

This study aims to improve the rheological properties of AL-Dura Refinery asphalt using cellulose acetate as a natural polymeric additive. The asphalt was mixed with cellulose acetate in various proportions (1, 2, 3, 4,5, and 6%), and the mixture was stirred at 150 °C for 60 min. The rheological properties of asphalt, on the other hand, were modified by using cellulose acetate in the presence of 1% sulfur, and the mixture was stirred for 60 min at 180 °C. The physical characteristics of the modified materials, such as ductility, softening point, and penetration index, were determined. For some of the models, the Marshall Quotient, aging, and chemical immersion tests were also performed. The best models were tested using IR and HNMR spectroscopy, as well as thermal measurements (Tga, Dta, DSC, Tg). The obtained models have rheological properties that make them suitable for a variety of applications, including paving, moisture barrier construction, and flattening. In addition, the modified asphalt is less affected by ageing conditions compared with the original asphalt, especially in terms of weight loss percentage. This is considered as a critical aspect of using cellulose acetate in the modification process.

Keywords: Cellulose acetate; Asphalt; Rheological properties; marshal; Thermo gravimetric analysis (TGA).

1. Introduction

The term Bitumen is used in Europe and is synonymous with the word (Asphalt) in North America, outside North America, the term asphalt is used to denote mixtures of bitumen with mineral materials [1]. It is a semi-liquid or solid substance of high viscosity with a dark brown color and a sticky produced as a waste of the crude oil distillation process [2-4]. Also, it can be described as very heavy crude oil that lost the bulk of its light volatile components, it can be described as a viscous distilled petroleum substance that contains a small percentage of volatile substances [5, 6]. Because of the urgent need to produce asphalt materials with rheological specifications used in paving, moisture-blocking materials, mastics and in various industrial fields, many researchers have improved the rheological properties of asphalt by various methods.

Ye et al. [7] have performed asphalt modification using cellulose fibers, polyester fibers, and mineral fibers as modifiers for the asphalt mixture. Xu et al. [8] studied the aging test on asphalt samples. Infrared spectroscopy was also used to study the effect of different ageing conditions on the performance of asphalt modified with lignin. They also studied the Dynamic Shear Rheometer test (DSR) and the Bending Beam Rheometer test (BBR). The results of the two tests showed that the addition of lignin positively affects the performance at high temperatures without negatively affecting the performance at low temperatures. Tang et al. [9] studied the rheological properties of asphalt modified with polymeric additives and sulfur. In their study, hybrid asphalt bonds containing crumb rubber, styrene-butadiene-styrene (SBS) and sulfur were prepared in different percentages. The results indicated that SBS and sulfur help to improve the high temperature properties and elastic behaviour of the asphalt bond, and the study recommended using 0.2 to 0.3% of sulfur. Pereze et al. [10] studied the addition of lignin as a natural polymer extracted from hardwood sheets to asphalt. Because polymers improve the performance of asphalt mixtures, this work studies the possibility of using this industrial waste as a bitumen extender in the production of asphalt mixtures. This study indicated resistance to humidity, permanent deformation and thermal exposure. The study concluded that it is appropriate to use industrial waste containing lignin in asphalt mixtures.

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Receive Date: 30 October 2021, **Revise Date:** 14 December 2021, **Accept Date:** 21 December 2021
DOI: 10.21608/EJCHEM.2021.103532.4790

Because polymers boost the effectiveness of asphalt mixtures, a large number of synthetic polymers were used in this field. In this paper, we use the cellulose acetate as a natural polymer for the modification of asphalt matrix. The physical, chemical, and thermal characteristics of the prepared models were investigated in order to demonstrate their performance in paving, moisture barrier, and flattening applications. The economic viability of this work is based on the fact that small amounts of cellulose acetate were used to improve the quality of the paving asphalt, resulting in an increase in the asphalt's operational life.

Experimental:

Al-Dura Asphalt was obtained from the Dura refinery, which has the specifications shown in **Table** 1. Anhydrous Aluminum Chloride ($AlCl_3$) and Sodium Carbonate were supplied by Fluka. Sulfur was Obtained from the General Company for Mishraq Sulfur and Cellulose acetate was supplied by British Drug Houses (BDH).

To know the best temperature for the mixing process, asphalt was treated with 1% of cellulose acetate in the presence of 1% of anhydrous aluminum chloride for one hour and at different temperatures for the purpose of determining the optimum temperature. **Table** 2 shows the results obtained.

For knowing the best time for the mixing process, asphalt was treated with 1% of cellulose acetate in the presence of 1% of anhydrous aluminum chloride with various time at the optimum temperature. **Table** 3 shows the results obtained.

After determining the optimum temperature and optimum time, the asphalt was treated with 1% cellulose acetate with different weight percentages of anhydrous aluminum chloride (0.120.25, 0.5, 1, 2, 3)% by weight at 150 °C for one hour for the purpose of determining the optimum ratio of anhydrous aluminum chloride and **table** 4 shows the results obtained.

The asphalt was treated with 1% cellulose acetate with different weight percentages of sulfur at a temperature of 180 °C for one hour [13]. **Table** 6 shows the results obtained for determining the optimum percentage of sulfur that can be used for subsequent modifications.

Cellulose acetate was used as a modified additive to asphalt according to the following pathways: In the first path, asphalt was treated with different percentages of cellulose acetate, ranging between 1-6 by weight, under the optimum conditions for the mixing process and in the presence of anhydrous aluminium chloride. The second path: the asphalt was treated with cellulose acetate in different proportions ranging from 1-6% by weight and in the presence of 1% by weight of sulfur at a temperature of 180 and a time of 60 minutes, which are the optimal conditions for the mixing process that was obtained.

The rheological specifications of the original and treated asphalt material were determined, including the measurement of ductility [14], Softening Point [15], Penetration [16] and calculating the penetration index for all models [17], as well as the marshal measurement [18] and aging [19] for some results. The IR and HNMR spectroscopy were measured. Thermal (Tga, Dta, DSC, Tg) of the models were also estimated.

Results and Discussion

Natural polymers are among the abundant and renewable materials available in nature. Therefore, our study focused on the use of these polymers in the field of obtaining a rheological modification of asphalt that makes it suitable for various uses, including in the field of paving. In our study, cellulose acetate was used as a modified material for asphalt. Cellulose acetate is a natural polymer made by reacting cellulose with acetic acid in the presence of concentrated sulfuric acid. Cellulose is a polysaccharide chemical compound extracted mostly from cotton and wood. Cellulose is the most prevalent natural polymer. A diagram of the preparation process can be introduced.

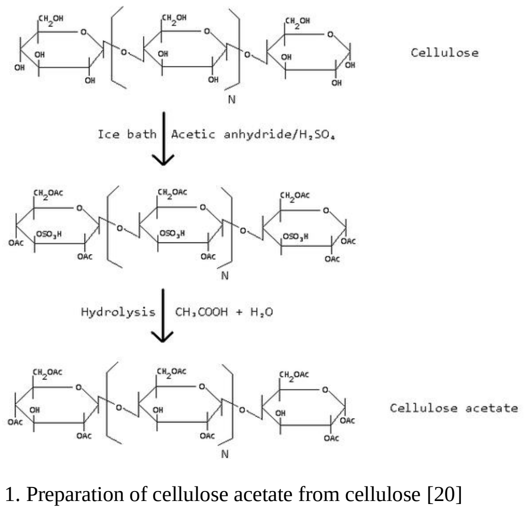

Fig. 1. Preparation of cellulose acetate from cellulose [20]

Initially, the optimal conditions for the process of mixing asphalt with cellulose acetate were determined. **Table** 2 indicates that using a temperature of 150 °C and a time of 60 min gave the best characteristics of asphalt, which is considered the optimum temperature to produce the required reaction and make rheological improvements. After determining the best temperature, the reaction time was varied in order to find the best time. The results obtained in **table** 3 show that the best time is 60 min.

After determining the optimal conditions for the mixing process, the percentage of anhydrous aluminum chloride added was changed, and **Table** 4 shows the results obtained. It is clear that the percentage that was adopted at the beginning of the practical part, which is 1% by weight of anhydrous aluminum chloride, did not represent the optimal percentage that could be used, and that the best percentage was 0.25% by weight of anhydrous aluminum chloride. Then the asphalt was treated with cellulose acetate under the optimum conditions for the mixing process and the percentage of anhydrous aluminum chloride added. **Table** 5 and **figure** 2 show the results obtained.

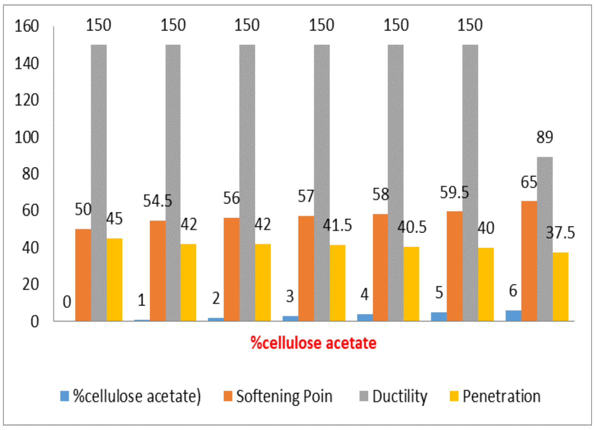

Fig. 2. Rheological properties of asphalt with cellulose acetate by weight in the presence of 0.25 % by weight of aluminum chloride at a temperature of 150 °C for 60 min

It is clear from **table** 5 that the use of cellulose acetate as a modified material was effective at percentages that do not exceed 5% by weight, according to the standard specifications shown in the **table** 5. It is also clear that the rheological properties were acceptable and good at less than 5%, which indicates an effective interaction between cellulose acetate and asphalt at these percentages of cellulose acetate under the conditions used.

In a second attempt to find another path to add cellulose acetate, the mixing process of the asphalt material was carried out in the presence of 1% of cellulose acetate with different percentages of sulfur in order to know the optimal conditions for adding cellulose acetate later. **Table** 6 shows the results obtained.

It is clear from the that the best percentage used is 1% by weight of sulfur. Heating asphalt with sulfur at 180 °C leads to the transformation of the eight sulfur rings into free radicals and then its association with asphalt and thus giving flexibility to the chains between which it enters [9].

The asphalt was then treated with cellulose acetate at 180 °C with ratio ranging between 1 to 6% by weight and in the presence of sulfur with 1% by weight.

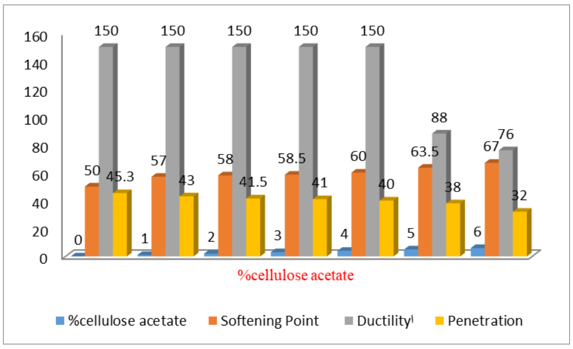

Fig. 3. Rheological properties of asphalt with cellulose acetate by weight in the presence of 1% by weight of sulfur

It is noted from **Table** 7 that, the results were all good and acceptable, with the best percentage at 4% of cellulose acetate in the presence of sulfur. And the rheological properties were within the standard specifications of asphalt paving shown in **Table** 1, and the models (31, 32) can be used as mastics, flattening and for roofs protection from water and moisture.

It was clear from these two tracks that, cellulose acetate could be used to modify the rheological properties of asphalt. The results were acceptable in both tracks. The presence of effective groups of cellulose acetate and the presence of $AlCl_3$ as a catalyst works to increase the bonding between cellulose acetate with the asphalt material, which in turn leads to an increase in the homogeneity of the asphalt system and an improvement in the rheological properties of asphalt. Almost the same thing happens in the case of sulfur when treating asphalt with cellulose acetate.

IR and H-NMR, spectroscopy were measured, as well as Thermal (Tga, Dta, DSC, Tg) for the models 0, 19.

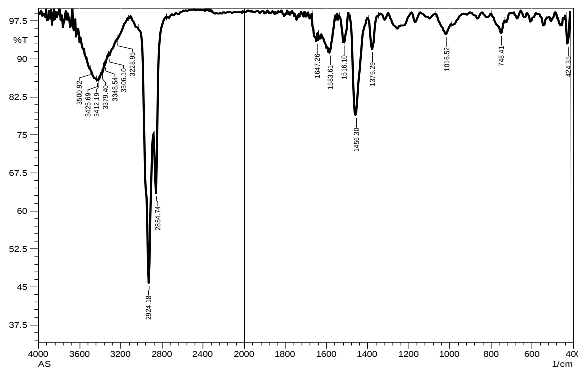

Fig. 4. FT-IR spectrum of the original asphalt

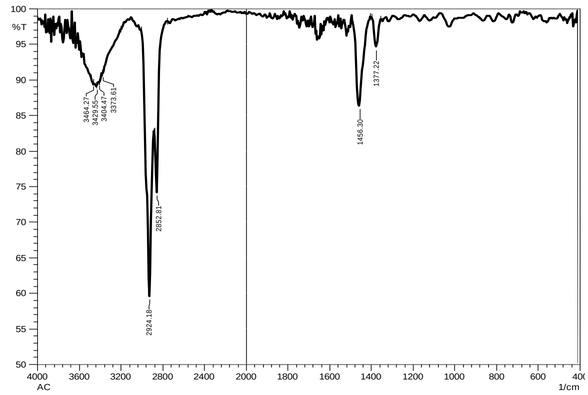

Fig. 5. FT-IR spectrum of asphalt treated with cellulose acetate

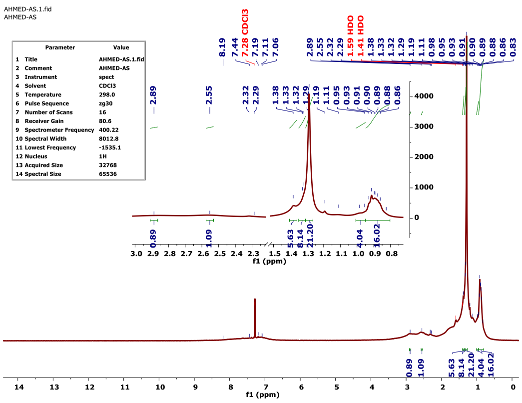

Fig. 6. H-NMR spectrum of parent asphalt

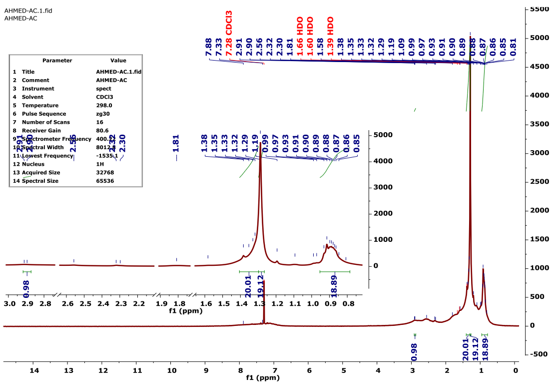

Fig. 7. H-NMR spectrum of asphalt treated with cellulose

Figure 4 represents the IR spectroscopy for the original asphalt. **Figure** 5, representing the asphalt treated with cellulose acetate. While **Figure** 6 shows the H-NMR spectrum of the original asphalt, and **Figure** 7, is the H-NMR spectrum of the treated asphalt with acetate. By comparison with the spectra of the previous models, it was found that there was no change in the OH band in the IR, meaning that there was no change in the polarity value of the asphalt treated with cellulose acetate. While when observing the H-NMR spectrum in **Figures** 6 and 7, it is noticed that there was a change in the intensity of the signal when cellulose acetate was added. In the aliphatic part. The probability of converting aliphatic to aromatic is weak, as evidenced by the fact that the signal of the alkyl part that appears at 1.5ppm did not show any significant change. Therefore, we believe that $AlCl_3$ did not contribute to the alkylation process. This is inferred from the lack of influence of the signal at 1.5ppm, and this indicates that the role of $AlCl_3$ is to contribute to the reforming process and the formation of the aromatic part, and this is evident from the intensity of the signal at approximately 7.4ppm.

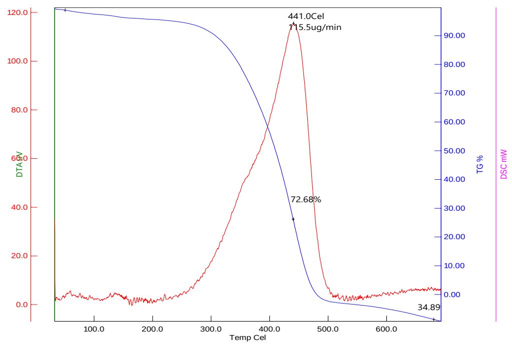

Fig. 8. Thermo gravimetric Analysis (TGA) of the original asphalt

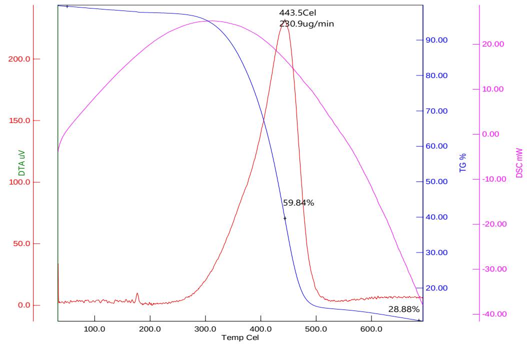

Fig. 9. Thermo gravimetric Analysis (TGA) for asphalt treated with cellulose acetate

Figures 8 and 9 represent the thermal analysis of the models (0 and 19). The figures were divided into four regions according to weight loss. The first zone range from 0 to 200 m, as the weight loss was at minimum, while the process of weight loss increased in the second zone, which is between 200-400 m. It was noticed in the figures, there was a steady increase in the third region, which is confined between 400-600°C, for weight loss at temperature 441°C for model (0) while it was at 443ºC for model (19) and the weight loss continued continuously in this region. There is also a slight weight loss in the fourth region, which is represented by temperature higher than 600 ºC. The fracture of the solid carbonaceous materials led to deterioration in the final region. This observation is consistent with that reported by Bach et al. [20]. The results of the thermal analysis of the samples showed that there was no significant change in the weight within the first region, which represents the conditions of preparation and uses of asphalt. The samples also showed weight loss at temperatures higher than 200°C, which are higher temperatures than required during preparation. This indicates the high stability of samples during preparation and during the period of use. It was also noted that the most weight loss occurred in the third region between 420-460 °C, while the weight loss decreased at a temperature of more than 490 °C. It was found that the weight loss of the model (19) was less than the model (0), and this indicates the high stability of the modified model.

The change in the weight loss and breakdown of asphalt is related to the components of the asphalt. Asphalt is composed of two malate (saturated, aromatic and resinous) and asphaltene. The weight loss of asphalt occurs through the decomposition and breakdown of the saturated and unsaturated maltene, followed by the aromatic compounds, and finally the asphaltene. This result was confirmed by a previous comprehensive study conducted by Shi et al. [21]. In fact, no significant deviation was observed between modified asphalt and virgin asphalt in view of the TG and DTG curves. Hence, it can be concluded that the modification by cellulose acetate was successful.

In order to test the suitability of asphalt models for paving purposes, the Marshall examination (paving asphalt) was conducted for the best model of the models obtained from the results of previous measurements as well as the original asphalt. This measurement gives an indication of the suitability of the asphalt to be used for paving by applying pressure on the model to be tested. Marshall Quotient from the product of Marshall Stability divided by the value of creep [19]. **Table** 8 shows the values of stability and creep for the best model obtained and compared with the original model according to the specifications of the Iraqi Roads and Bridges Authority (SCRB) [22].

In order to know the extent to which modified asphalt models are affected by the aging conditions, a furnace examination was conducted for thin asphalt films **table** 9.

Table 9 indicates that, the degree of influence of the modified asphalt with the aging conditions of temperature and oxygen in general is less than the degree of influence of the original asphalt, especially the percentage of weight loss. This inferred that, the modified asphalt model is characterized by great resistance to stress and fewer cracks, as well as a long operational life. This is considered a positive thing for modification with naturally modified polymers.

Table 1 : The specifications of Al-Dura refinery asphalt

Test type	Test results	American Standard No [11]	Iraqi Standard Specifications [12]
Penetration (25 ºC, 100 g, 5 sec, 0.1mm)	45.3	40-50	40 – 50
Ductility (25ºC, 5 cm/min, cm)	150+	+100	100+
Softening Point (5ºC/min, ºC)	49	30-157	49 – 58
Specific Gravity (25 ºC)	1.053	....	1.05- 103
Flash Point	277	…..	232≥
Loss on Heating (5 hr, 163ºC), %	0.36	0.2	……….
Solubility in ethylene trichloride	99.7	99.9	....
Asphaltene percentage	18.3	....	……

Table 2: Rheological properties of asphalt with 1% cellulose acetate in the presence of 1% anhydrous aluminum chloride for one hour

Sample	Temp.ºC	Ductility (25ºC, 5 cm/min, cm)	Softening Point (5ºC/min, ºC)	Penetration (25 ºC, 100 g, 5 sec, 0.1mm)	Penetration index
AS0	0	150	50	45.3	-1.413
AS1	120	150	52	41	-1.150
AS2	150	150	53	42	-0.695
AS3	170	125	52.5	39	-1.137
AS4	180	110	52	38	-1.84
AS5	190	88	53	35	-1.243
AS6	200	83	51	33	-1.809

AS0 original model without any transaction

Table 3: Rheological properties of asphalt with 1% cellulose acetate in the presence of 1% anhydrous aluminum chloride at 150 ºC

Sample	Time (min)	Ductility (25ºC, 5 cm/min, cm)	Softening Point (5ºC/min, ºC)	Penetration (25 ºC, 100 g, 5 sec, 0.1mm)	Penetration index
AS0	0	150	50	45.3	-1.413
AS7	30	150	50	44	-1.150
AS2	60	150	٥٣	42	-0.695
AS8	90	110	54	39	-1.137
AS9	120	100	55	38	-1.84

Table 4: Rheological properties of asphalt treated with 1% by weight of cellulose acetate and different ratio of the catalyst at a temperature of 150 ° C and a time of 60 min

Sample	%catalyst	Ductility (25ºC, 5 cm/min, cm)	Softening Point (5ºC/min, ºC)	Penetration (25 ºC, 100 g, 5 sec, 0.1mm)	Penetration index
AS0	0	150	50	45.3	-1.413
AS*	0	122	52	44	-1.116
AS10	0.125	138	٥٣	43	-0.701
AS11	0.25	150	٥٢,٥	43	-0. 682
AS12	0.5	150	٥٢	41	-0.683
AS2	1	150	٥٣	42	-0.867
AS13	2	68	٥١	35	-0.391
AS14	3	62	59	32	-0.18

*AS is an oxidizing form without a catalyst at optimum conditions

Table 5: Rheological properties of asphalt treated with different percentages of cellulose acetate and in the presence of 0.25 of aluminum chloride at a temperature of 150 °C for 60 min

Sample	%cellulose acetate	Ductility (25ºC, 5 cm/min, cm)	Softening Point (5ºC/min, ºC)	Penetration (25 ºC, 100 g, 5 sec, 0.1mm)	Penetration index
AS0	0	150	50	45.3	-1.413
AS15	1	150	54.5	41	-0.533
AS16	2	150	56	42	-0.266
AS17	3	150	57	39	-0.082
AS18	4	150	58	38	-0.097
AS19	5	150	59	35	-0.351
AS20	6	89	65	33	1.273

Table 6: Rheological properties of asphalt treated with 1% by weight of cellulose acetate and different percentages of sulfur

Sample	%Sulfur	Ductility (25ºC, 5 cm/min, cm)	Softening Point (5ºC/min, ºC)	Penetration (25 ºC, 100 g, 5 sec, 0.1mm)	Penetration index
AS0	0	150	50	45.3	-1.413
AS21	0.25	150	54	45	-0.491
AS22	0.5	146	54.5	44	-0.43
AS23	0.75	١48	55.5	43	-0.262
AS24	1	150	57	42	0.0
AS25	2	85	63	35	0.76
AS26	3	78	67	34	1.393

Table 7: Rheological properties of asphalt treated with 1% by weight of sulfur and different percentages of cellulose acetate

Sample	%cellulose acetate	Ductility (25ºC, 5 cm/min, cm)	Softening Point (5ºC/min, ºC)	Penetration (25 ºC, 100 g, 5 sec, 0.1mm)	Penetration index
AS0	0	150	50	45.3	-1.413
AS27	1	150	57	41	0.0
AS28	2	150	58	42	0.183
AS29	3	150	58.5	39	0.252
AS30	4	150	60	38	0.496
AS31	5	88	63.5	35	1.033
AS32	6	76	67	33	1.261

Table 8: Stability and creep values of the parent and modified asphalt with cellulose acetate and specifications of the Roads and Bridges Authority (S.C.R.B)

Sample No.	%of asphalt added to cumulus	Modified asphalt (best models)		
		Stability (KN)	Creep (mm)	MQ
AS0	4.75	11.3	4.1	2.75
AS19		13.2	2.41	5.47
AS**	4-6	7 minimum	2-4	3.5 minimum

Table 9: Rheological properties of original asphalt and cellulose acetate-based asphalt before and after they were subjected to the asphalt thin film oven test (TFOT)

Sample	Type of sample	Rheological properties	Before test	After test	Difference
AS0	Origin	Ductility (25ºC, 5 cm/min, cm)	150	…..	
		Softening Point (5ºC/min, ºC)	50	53	3
		Penetration (25 ºC, 100 g, 5 sec, 0.1mm)	45.3	42.1	3.2
		Penetration index (PI)	-1.413	-0.863	0.55
		Weight of loss%	…..	0.05	
AS19	Modified by cellulose acetate	Ductility (25ºC, 5 cm/min, cm)	150	141	4
		Softening Point (5ºC/min, ºC)	59	61	2
		Penetration (25 ºC, 100 g, 5 sec, 0.1mm)	41	40.3	0.7
		Penetration index (PI)	0.351	0.706	-0.355
		Weight of loss%	……	0.02	

2. Conclusions

Addition of cellulose acetate to the asphalt affected the rheological properties of the asphalt system in varying proportions. The process of adding cellulose acetate as an additive to asphalt in the presence of anhydrous aluminum chloride significantly improved the rheological properties of asphalt when compared with the original asphalt. Where all percentages were acceptable and good at less than 5%. The process of adding cellulose acetate as an additive to asphalt in the presence of sulfur improved the rheological properties of asphalt in an excellent way when compared with the original models on it, as all proportions were acceptable and good at less than 4%. This study gave better Marshall values for the modified model with cellulose acetate than in the original asphalt, and this indicates the possibility of using this additive in tiling operations in a way that suits the atmosphere and climate of our country. This study indicated that the modified models with cellulose acetate are less affected by the ageing conditions compared to the original asphalt. As shown by the study and through thermal decomposition measurements, cellulose acetate is suitable as an additive to modify and improve the rheological properties of asphalt.

3. Conflicts of interest

There are no conflicts to declare.

4. Acknowledgments

The researchers express their thanks and appreciation to the Department of Chemistry at the College of Education for Pure Sciences - University of Mosul. And to the College of Engineering - Civil Department, University of Mosul to provide the necessary facilities to complete this research

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