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Case study

Mechanical properties of asphalt concrete modified with carbon nanotubes (CNTs)

Mohamed Samir Eisa [a], Ahmed Mohamady [b], Mohamed E. Basiouny [a], Ayman Abdulhamid [a], Jong R. Kim [c,*]

[a] Department of Civil Engineering, Benha Faculty of Engineering, Benha University, Benha, Egypt
[b] Department of Construction Engineering and Utilities, Faculty of Engineering, Zagazig University, Zagazig, Egypt
[c] Department of Civil and Environmental Engineering, School of Engineering and Digital Sciences, Nur-Sultan, Kazakhstan

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ABSTRACT

Three different percentages of carbon nanotubes (CNTs) (0.1%, 0.5%, and 1% by mass of asphalt cement) were used to modify conventional asphalt cement (60/70) in this study. Mechanical properties of modified asphalt cement and mixture were evaluated. Penetration grade, kinematic viscosity, softening point and dynamic shear rheometer test were measured to evaluate physical properties of modified asphalt cement. The results exhibited that modifying asphalt cement with CNTs decreased its penetration and increased its kinematic viscosity and softening point. Rutting parameter increased with CNTs at the given temperature for both unaged and RTFOT-aged samples. Marshall stability tests, low temperature cracking tests, indirect tensile tests and wheel tracking tests were conducted to assess the mechanical performance of modified hot asphalt mixture. The Marshall stability increased with CNTs but no significant difference at 0.5 and 1.0 wt%, while Marshall flow decreased with CNTs. The results of wheel tracking test showed that rut depth decreased by 45% upon adding 0.5% CNTs by weight of asphalt cement; also, this percentage of CNTs endowed improvement in low temperature cracking and indirect tensile strength of the asphalt concrete. This study underlines that adding CNTs into asphalt cement enhances the performance of asphalt concrete pavement in both hot and cold weather, which in turn prolongs the pavement's service life and saves maintenance expenses.

1. Introduction

Roads are considered the pivotal arteries of the development of a nation. The annual consumption of bitumen in Egypt is ~1.5 million tons per year in a very massive budget. A large portion of this amount is for rehabilitation of existing roads network. The major reasons for rapid deterioration in highways are rutting and fatigue cracking in the pavements. It is believed that the properties of the binders and aggregates used in the asphalt mixtures play crucial roles in the pavement performance. Binders are commonly modified to meet performance requirements for wide range of loads and temperatures. In the last few decades, the fast growth in population and development of cities have led to considerable rise in traffic volume and vehicles loads. As a result, traditional pavements are not able to reach the end of their life span due to severe fatigue cracking and permanent deformation (rutting). Therefore, criteria and standards for building new highways and transportation infrastructure are in urgent need for update.

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* Corresponding author.
E-mail address: jong.kim@nu.edu.kz (J.R. Kim).

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Bitumen is the most common binder in hot asphalt mix; it is a viscoelastic material where temperature and rate of loading have marked impact on its performance. At high-temperature, pavement distresses are not able to preserve its original shape under traffic loads leading to permanent deformation while bitumen loses its elasticity at low-temperature and begins to crack due to resulted stiff structure. These phenomena reveal that unmodified bitumen cannot endure the increase of the traffic volume and load and has limited ability to face wide range of temperatures over the service life of the pavement. Therefore, modifying bitumen is essential to overcome the aforementioned limitations and enhance mechanical performance of the asphalt concrete using additives such as polymers and inorganic fillers [1–4]. However, it is always a challenge to select the most suitable modifying agent for the asphalt to satisfy rutting resistance and resilience. Since they are conflicting properties of a material, there has always been a trade-off between rutting resistance (at high temperature) and fatigue resistance (at low temperature) for the asphalt concrete upon adding modifying agent. Therefore, based on the service and temperature where the asphalt concrete is applied, engineers define the modifier.

Modifying asphalt binders enhances adhesion, temperature sensitivity, friction characteristics, aging and durability of asphalt concrete. Traditional modifiers may not attain the required properties in asphalt concrete to sustain diverse traffic loads and climate change [5]. Recently, there has been an emerging interest in applying nanofillers in asphalt modification aiming at high mechanical performance highways [6,7]. Nanofillers feature outstanding surface area along with novel physical properties – a feat unmatched by other materials. Outstanding surface area of nanofillers leads to short filler-to-filler distance and strong interface with bituminous matrix. These advantages result in good mechanical properties and durable asphalt mix at low fractions (0.5–1.5 wt%). A plethora of research has been conducted on modifying bituminous binders by various nanofillers such as nanoclay [8–10], nanosilica [11,12], titanium dioxide nanoparticles [13,14], nanohydrated lime [15] and nanofibers [16,17]. Yao et al. [18] conducted a study on bitumen modification using nanosilica at 4% and 6% by bitumen weight to evaluate the characteristics of modified asphalt. Nanosilica has been shown to enhance anti-aging property, rutting and fatigue cracking performance. Notably, the addition of nanosilica to asphalt mixture significantly enhances its dynamic modulus, flow number and rutting resistance in comparison with unmodified asphalt mixtures.

Polymeric materials such as styrene-butadiene-styrene block copolymer (SBS) and ethylene vinyl acetate and styrene butadiene rubber were commonly used to modify bitumen. Recently, there have been many studies that combine the soft nature of polymeric additives with outstanding properties of nanofillers to synergistically improve the properties of bitumen and its hot mix. Organic nanoclay (Palygorskite) modified by SBS was successfully added to bitumen resulting in high quality asphalt concrete; water susceptibility was improved and critical temperature increased by 40% upon addition of 3 wt% nanoclay-SBS to asphalt [19]. In another study, Sayed A.G. et al. [20] reported synergy between nanoclay, nanolime and SBS when added together to modify asphalt with optimum combination percentages at 4 wt% nanoclay, 6 wt% nanolime and 3 wt% SBS.

Carbon-based nanomaterials, namely carbon nanotubes (CNTs) and graphene possess outstanding intrinsic properties; their Young's moduli and tensile strengths are ~1 TPa and $50-130$ GPa, respectively. Electrical and thermal conductivity of graphene are 6000 $\text{S}\cdot\text{cm}^{-1}$ and 5000 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$, in comparison with the values of $100-10^6$ S·cm-1 and ~3000 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ for CNTs [21]. CNTs and graphene have been extensively studied for the development of multifunctional polymer nanocomposites to create high mechanical performance, electrical and thermal conductivity, barrier performance and recently to greatly reduced flammability [22,23].

The application of CNTs in asphalt modification for roads paving has enticed researchers recently to extend more effort in this area. Xiao et al. [24–26] showed that CNTs improved both physical and workability properties of bitumen as well as the mechanical performance of hot asphalt mix. Complex modulus, elastic modulus and viscous modulus have been observed to increase upon modifying bitumen with CNTs [27]. CNTs at 1.5 wt% increased storage modulus of asphalt binder by two folds – from ~15 to ~30 MPa at 100 Hz [27]. More importantly, adding 3 wt% of CNTs to bitumen decreased rut depth of hot mix asphalt by 37% at 40 °C [28]. El-badawy's group [29] reported that MWCNTs enhanced the viscoelastic behaviour (complex storage modulus and phase angle) of bitumen predicting adequate rutting resistance. Amir K. et al. [24] and Ameri et al. [27] reported that the resistance of bitumen for permanent deformation and fatigue were significantly improved by adding CNT. Mean fatigue life of asphalt mix was increased by 287% at 1.5 wt % CNTs dosage [27]. Fracture test results of asphalt hot mix showed remarkable enhancements in fracture strain energy upon modifying asphalt binder with CNTs; 1.5 wt% CNTs increased the strain energy to failure from 0.145 to 1.752 N.m of the asphalt mixture [27].

Not only did fatigue resistance improve by adding CNTs into bitumen but also its self-healing property improved as noticed by Santagata et al. [30,31]. Creating and improving synergy between additives is of great interests to the pavement industry in addressing various limitations; SBS block copolymer enhanced the interface between multiwalled CNTs and bitumen [32]. Also, the study [32] showed that molecular structure of SBS modifier has a crucial role as a compatibilizer between CNTs and bitumen; linear SBS helped in forming a stable and continues interface between CNTs and bitumen.

Despite the published work on bitumen modified by nanoadditives, the current knowledge on how the nanoadditives affect the overall performance of asphalt mixture is still in its infancy. The study presents more insights into application of CNTs in the pavement industry mainly to enhance rutting resistance at high temperature and hinder crack propagation at low temperature. The literature has scarcely studied the direct effect of carbon-nanomaterial including CNTs and graphene on the rutting deformation of asphalt mixtures. Specifically, our current study aims to (i) investigate the effect of CNTs on the rheological and physical properties of bitumen (binder); (ii) select the optimum fraction of CNTs to prepare hot mix asphalt modified with CNTs; and (iii) investigate mechanical performance of hot mix asphalt including rutting resistance, indirect tensile strength and low temperature cracking test. It is noteworthy that wheel tracking test was conducted to perform rutting resistance; wheel tracking test is a field-like test which gives solid evidence on permanent deformation resistance upon adding CNTs. Moreover, the study investigated the initial cost of modifying asphalt mixtures by CNTs.

2. Materials and methods

The asphalt binder (bitumen) of 60/70 penetration grade was purchased from Suez Refinery Company, Egypt. All aggregates including coarse aggregates, sand and mineral fillers were kindly provided from the Egyptian Transportation Organization. Multi-walled carbon nanotubes (CNTs) were used as a modifier for the asphalt binder. Industrial grade multi-walled CNTs were provided by Jiangsu XFNANO Materials Tech Co. Ltd, China. Properties of CNTs were as follows: 95% in purity, outer diameter 10–30 nm, inner diameter 5 −10 nm, tube length 10–30 µm, specific surface area of 110 m^2/g and actual density of 2.1 g/m^3. All materials used in this work were used as received without further purifications.

2.1. Aggregate

Two types of aggregates: coarse and fine aggregates from Suez quarry were used to produce hot mix asphalt. Crushed limestone aggregate was used in this study as coarse aggregate and its powder was used as mineral filler. Coarse aggregate passed at least 70% through sieve of size 19 mm while the aggregates within size range of 2.36 mm — 75 µm represented the fine aggregates. The fine aggregate was siliceous sand in all designed asphalt concrete mixes that has had a specific gravity of 2.673. The design gradation of aggregate used to produce wearing surface asphalt course was 4 C gradation. The properties of the aggregates are summarized in Table 1.

2.2. Bitumen

Since bitumen type affects the properties of the produced asphalt mixture, asphalt binder used in this study was obtained from one source, Suez asphalt cement. In this work bitumen with 60/70 penetration grade, PG64–22, was used; its physical properties are presented in Table 2.

2.3. Bitumen/CNT composite preparation

The main objective of the work is to study and investigate the improvements in properties of modified bitumen upon the addition of CNTs and its hot mix asphalt. The conventional bitumen was liquified by heating it up to 150 °C [33,34]. Then, CNTs were added and mixed with bitumen using high shear mechanical mixer for one hour at 150 °C to obtain good dispersion. Three different percentages of CNTs namely, 0.1%, 0.5%, and 1% by mass of bitumen were investigated. It is understood that heating to high temperature and stirring for long time might have an aging effect on bitumen. Therefore, two control samples were prepared to measure the effect of mixing conditions on the bitumen, both are at 0 wt% CNTs: (i) as-received sample which did not experience heating or stirring; and (ii) processed sample which was exposed to mixing conditions of 150 °C and 1 hr stirring.

2.4. Asphalt mixture design

In this study, the technical specifications of Egyptian Transportation Organization for Highway and Bridges Construction were followed. Crushed lime-stone aggregates, sand, and filler were graded in laboratory sieves to fall within the upper and lower limits, conforming with the Egyptian Code of Practice for urban and rural roads (ECP, 2008) [35]. Fig. 1 presents the aggregate gradation used in the current study for wearing surface layer (heavy traffic volume). Marshall design method [36] was used to determine the optimum bitumen content in all mixtures according to ASTM D6927 [37]. Accordingly, bitumen percentage was set at 5.2 wt% and air voids was measured as 3.8%. Since CNTs were added at very small weight percentage; all prepared asphalt mixtures comprised of same gradation and optimum bitumen content.

2.5. Characterization and measurements

Various number of tests were carried out to investigate the effect of CNTs on the properties of bitumen and hot asphalt mix. First set of tests was penetration test, kinematic viscosity test and softening point test; they were associated with standards AASHTO T49 [38], ASTM-D4402 [39] and ASTM-D36/D36M [40], respectively. At least 3 replicates were prepared and tested; average of the measured property and standard deviation were calculated.

Table 1
Aggregate properties.

Property	AASHTO Designation No.	Value	Specification range
Coarse aggregate			
Specific Gravity	T-85	2.746	2.5–2.8
Water absorption %	T-85	3.5	≤ 5
Los Angeles Abrasion %	T-96	25	≤ 40
Fine aggregate			
Specific Gravity	T-84	2.673	
Water absorption %	T-84	2.6	≤ 3

Table 2
Physical properties of neat bitumen.

Property	Value	Test method	Specification range
Penetration (@ 25 °C), 0.1 mm	67	ASTM D5	60–70
Softening point, °C	51	ASTM D36	45–55
Viscosity@ 135 °C, cP	422	ASTM D4402	–
Specific Gravity	1.020	ASTM D70	1–1.1
Flash point, °C	270	–	≥ 250

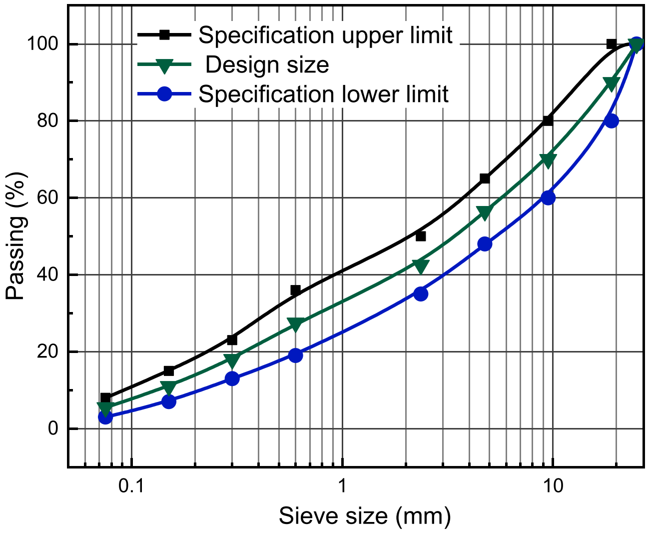

Fig. 1. Aggregate gradation of mix design.

Marshall stability and flow tests were conducted on all hot asphalt mixes in order to define the correct CNTs content within the investigated fractions in comparison with the unmodified asphalt concrete. Wheel tracking, indirect tensile strength and thermal stress restrained specimen tests were performed on the specimens at CNT content of 0.5 wt%. The results were compared to those from unmodified asphalt mixtures (0 wt% CNTs).

Dynamic shear rheometer (DSR) test was performed to measure complex modulus (G^*) and phase angle (δ). DSR test was carried out following standard AASHTO T 315–12 [41]. A 1-mm thickness film of bitumen was placed between two circular plates of 25 mm in diameter. The upper plate was oscillating at frequency 10 rad/sec while the lower plate was fixed. Two types of samples were tested:

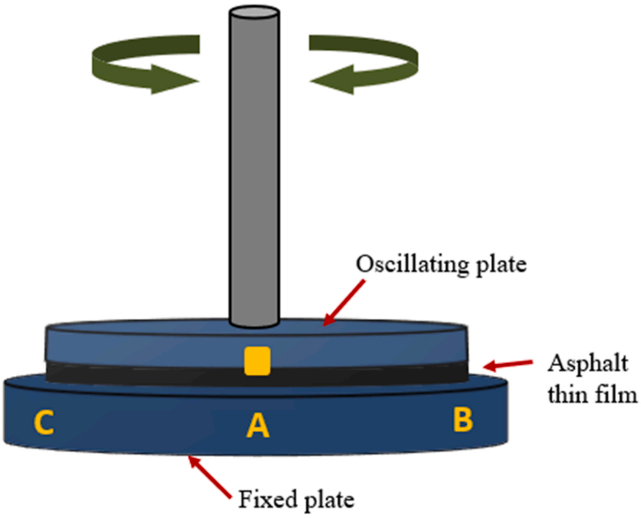

Fig. 2. A schematic diagram for Dynamic shear rheometer.

unaged and short-term aged samples. Rolling thin-film oven (RTFO) test was followed to prepare short-term aged sample according to ASTM D1754 [42]. A schematic diagram of DSR test is presented in Fig. 2. Samples at 0 wt%, 0.1 wt%, 0.5 wt% and 1 wt% CNTs-modified bitumen were tested on DSR for unaged and RTFO-aged conditions. The test was performed at temperatures 58, 64, 70 and 76 °C.

Wheel-tracking test is a key test to evaluate the rutting resistance of the asphalt mixtures [43]. The samples of asphalt concrete were subjected to wheel passes of 10,000 cycles. The sample was of slab shape with dimensions 265 × 320 × 40 mm according to standard AASHTO T-324 [44]. Firstly, the sample was compacted in a preheated mould. After 3 days, the sample was kept at 60 ℃ for 6 hrs. Secondly, the sample was moved to the wheel-tracking test machine as shown in Fig. 3. The test was conducted at 60 ℃ and continued till 10,000 cycles using a 70 kg-loaded wheel at rate of 53 passes/min (26.5 rpm). Rut gauge of 0.001 mm precision was used to measure the rut depth.

The thermal stress restrained specimen test used to evaluate the resistance of asphalt concrete against thermal cracking according to EN 12697–46 standard [45]. The TSRST device consists of environmental chamber, cooling device, screw jack, load frame, temperature controller, and computer data acquisition with control system. In TSRST, asphalt concrete specimen of dimensions 35 × 35 × 210 mm was glued by epoxy to two aluminium plates. Then the specimen was cooled down from room temperature at a rate of 10 ℃/hr; the length of the samples was constrained so that it was kept constant. The cooling of the specimen generates an induced stress which gradually increases with the cooling process. The induced stress results in specimen failure when the stress reaches its highest value and exceeds the specimen strength. The basic readings from the test are fracture temperature and fracture strength.

The indirect tensile strength (*ITS*) test was applied to assess tensile properties of the asphalt concrete thus, it is a direct indicator for crack resistance of the asphalt pavement. The tensile properties of asphalt concrete mixtures were measured by using Marshall-designed specimens; they were subjected to a compressive load along a diametric plane at constant rate of 51 mm/min. The compressive load acted vertically parallel to the diametrical plane of the sample through two loading strips as presented in Fig. 4. The load developed a tensile stress which acted uniformly perpendicular to the load direction through the diametrical plane. This would result in a failure along the vertical diameter of the specimen. The specimen diameter of 101.6 mm and height of 50.8 mm; plywood loading strips with width of 13 mm were used to produce relatively uniform stress distribution. The test was carried out at 25 ℃ based on ASTM D 6931 [46]. The peak value of the load causing failure was recorded to determine the tensile strength by using the relation $ITS = 2\,P_{max} / \pi\, t\, D$, where, *ITS* and P_{max} are the indirect tensile strength in MPa and the load-to-failure reached by the device in N, respectively; t and D are respectively the specimen thickness and diameter before the test, in mm.

Furthermore, statistical analysis was conducted to assess the effect of CNTs on rheological properties of bitumen and mechanical performance of hot mix asphalt. Specifically, analysis of variance (ANOVA) test was performed to show if CNTs significantly affect penetration, softening point and kinematic viscosity of bitumen, and Marshall stability, flow, indirect tensile strength, and fracture cryogenic properties of hot mix asphalt. ANOVA test was caried out using four levels of CNTs (0, 0.1, 0.5 and 1 wt%) with a confidence level of 95% and three replicates; results are presented in Tables 3, 4, and 6–8. Moreover, the normality of the residuals was tested using the normal probability plots; they are presented in the supplementary file to confirm the validity of ANOVA as a suitable statistical tool to analyse the recorded measurements.

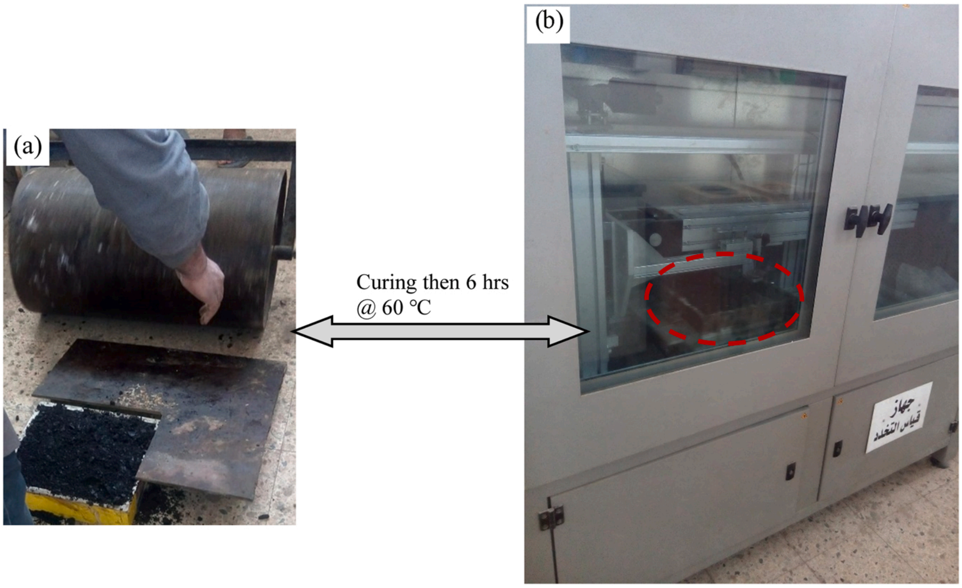

Fig. 3. Wheel tracking test preparation (a) compacting slab of asphalt concrete and (b) Hamburg wheel-tracking testing machine loaded with the sample.

Fig. 4. Indirect tensile tester with a loaded sample.

Table 3
ANOVA results for the rheological measurements.

	Source	DF	SS	MS	F-value	p-value
Kinematic viscosity	x	3	6811.3	2270.44	37.95	0.0001
	Error	8	478.7	59.83		
	Total	11	7290.0			
Penetration @ 25 ℃ (0.1 mm)	x	3	127.58	42.528	22.19	0.0001
	Error	8	15.33	1.917		
	Total	11	142.92			
Softening point	x	3	14.896	4.9653	10.36	0.004
	Error	8	3.833	0.4792		
	Total	11	18.729			

DF: Degrees of freedom **SS:** Sum of squares **MS:** Mean squares value

Table 4
ANOVA results for Marshall stability and flow.

	Source	DF	SS	MS	F-value	p-value
Marshall stability	x	3	73,923	24,641.0	142.64	0.001
	Error	8	1382	172.7		
	Total	11	75,305			
Flow	x	3	0.9622	0.32074	13.35	0.002
	Error	8	0.1923	0.02403		
	Total	11	1.1545			

DF: Degrees of freedom **SS:** Sum of squares **MS:** Mean squares value

Table 5
Low temperature cracking test results for unmodified and CNTs-modified asphalt mixtures.

Asphalt concrete sample-modified	Fracture cryogenic stress (MPa)	Fracture temperature (℃)	Cryogenic stress @ −20 ℃ (MPa)
0 wt% CNTs	4.3 ± 0.10	-24.8 ± 0.21	2.75 ± 0.04
0.1 wt% CNTs	4.36 ± 0.06	-25.3 ± 0.38	2.61 ± 0.03
0.5 wt% CNTs	4.55 ± 0.06	-26.4 ± 0.58	2.55 ± 0.08
1.0 wt% CNTs	4.56 ± 0.04	-26.2 ± 1.05	2.52 ± 0.06

Table 6
ANOVA results for low temperature cracking test.

	Source	DF	SS	MS	F-value	p-value
Fracture cryogenic stress	x	3	0.16513	0.055044	12.49	0.002
	Error	8	0.03527	0.004408		
	Total	11	0.20040			
Fracture temperature	x	3	5.252	1.7508	4.28	0.044
	Error	8	3.273	0.4092		
	Total	11	8.526			
Cryogenic stress @ −20 ℃	x	3	0.09386	0.031288	9.24	0.006
	Error	8	0.02710	0.003387		
	Total	11	0.12096			

DF: Degrees of freedom **SS:** Sum of squares **MS:** Mean squares value

Table 7
ANOVA results for indirect tensile test.

	Source	DF	SS	MS	F-value	p-value
Indirect tensile strength	x	3	73,923	24,641.0	142.64	0.001
	Error	8	1382	172.7		
	Total	11	75,305			

DF: Degrees of freedom **SS:** Sum of squares **MS:** Mean squares value

Table 8
Analysis of Variance (ANOVA) results for wheel tracking test.

	Source	DF	SS	MS	F-value	p-value
Rut depth @ 5000 cycle	x	1	14.602	14.6016	56.60	0.002
	Error	4	1.032	0.2580		
	Total	5	15.634			
Rut depth @ 10,000 cycle	x	1	20.2658	20.2658	198.00	0.001
	Error	4	0.4094	0.1024		
	Total	5	20.6752			

DF: Degrees of freedom **SS:** Sum of squares **MS:** Mean squares value

3. Results and discussion

3.1. Physical properties of bitumen

The effect of CNTs on physical properties of modified bitumen was investigated via tests including penetration @ 25 ℃ (0.1 mm), softening point, and kinematic viscosity tests; the test results are presented in Fig. 5a–c. The ANOVA test results at four levels of CNTs (0 @150 ℃ and 1hr-stirring, 0.1, 0.5 and 1 wt%) with three replicates of each response (i.e., penetration, softening point or kinematic viscosity) are presented in Table 3. Boxplots of penetration, softening point and kinematic viscosity are presented in Fig. S1–S5 in the supplementary file. The ANOVA results in Table 3 show that the CNTs significantly affect kinematic viscosity, softening point and penetration as the corresponding p-values are less than 0.05. The softening point and kinematic viscosity increased with the addition of CNTs whereas the penetration decreased. Fig. 5a shows that 0.1 wt% CNTs has marginal effect on the bitumen properties in comparison with the control samples at 0 wt%. As the CNTs content increases, the modification effect became noticeable; at 1 wt% CNTs the kinematic viscosity of bitumen increased by 13.5% in comparison with the control sample processed at 150 ℃ for 1 hr. This is a direct indicator that CNTs-modified bitumen would have higher resistance to deformation (rutting) in comparison with the unmodified sample. However, high bitumen viscosity means high mixing and compacting temperature which in turn would deteriorate its aging resistance. According to AASHTO T-316 [47], the viscosity values obtained in this study are within the permissible limit.

In general, the penetration grade decreases with the addition of stiff fillers [48]. Adding 1 wt% of CNTs decreased the penetration grade by ~ 13% in comparison with the processed control sample. The significant decrease of the modified bitumen penetration reduces the chance of rutting process. This result is important in road pavement in hot climate countries. Softening point of bitumen was increased by 4.8% at 1 wt% of CNTs which was in accord with the decrease of penetration grade. The decrease in penetration grade and increase of softening point upon modifying asphalt by CNTs would benefit the hot asphalt mix by having adequate deformation resistance at high temperature and heavy traffic load. It is noteworthy to mention that heating at 150 ℃ and stirring for 1 hr did not significantly affect the properties of bitumen. The kinematic viscosity, softening point and penetration of the as-received bitumen were close to those of the processed unmodified bitumen. Therefore, the effect of adding CNTs into bitumen was dominant in comparison with the hardening (aging) due to mixing at 150 ℃ for 1 hr. This was also concluded in previous study [49] where there was no significant difference in viscosity, complex modulus and creep stiffness over temperature sweep testing within the range of

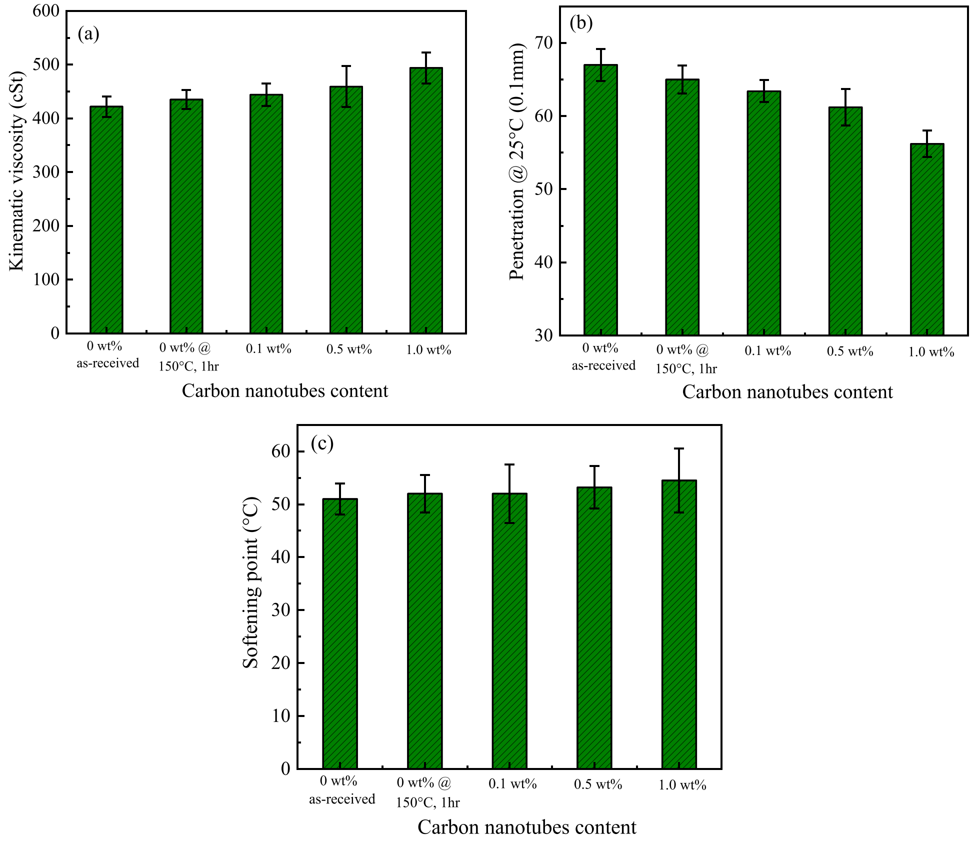

Fig. 5. Physical properties of unmodified bitumen and CNTs-modified bitumen: (a) kinematic viscosity, (b) penetration level at 25 ℃ @ 0.1 mm and (c) softening point.

35–110 ℃ at frequency of 10 rad/s [49]. Subsequently, we used as-received bitumen to prepare the control sample in the following tests.

3.2. *Rheological properties of bitumen and its CNTs-based composites*

Rut parameter of unmodified and modified bitumen with CNTs for three mass percentages 0.1%, 0.5%, and 1% are presented in Fig. 6a & b for unaged and RTFO- samples, respectively. Rut parameter limits were set at 1 kPa and 2.2 kPa for unaged and RTFO-samples, respectively. It is evident that rut parameter consistently decreases with the increase in temperature for all samples; for example, at 0.5 wt%, RP dropped from 6.1 kPa to 1 kPa (84%) for unaged sample when the temperature was elevated from 58 ℃ to 76 ℃; in case of RTFO sample, RP decreased by 88% within the same temperature range. It is clear that all samples have rut parameter higher than boundary limits for unaged and aged conditions at 64 ℃ (64 grade). At 70 ℃, unmodified bitumen and 0.1 wt% CNT-

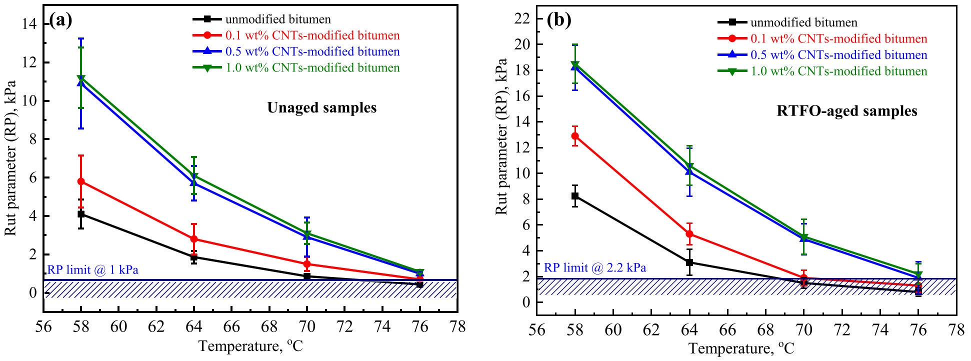

Fig. 6. Rut parameter (RP) of (a) unaged samples and (b) RTFO-aged samples of unmodified bitumen and its CNT nanocomposites.

modified bitumen could not withstand the applied shear stress and the RP fell underneath 2.2 kPa limit of RTFO-ageing conditions; 0.1 wt% CNT-modified unaged sample was successful in reaching grade 70.

Though all samples did not satisfy the boundary limit of RP at high temperature such as 76 ℃, it was evident that CNTs promoted the rut parameter of all samples at various conditions within temperature range of 58–70 ℃; for example, 1 wt% CNTs increased the rut parameter by 173% and 124% at 58 ℃ for unaged and RTFO sample, respectively, in comparison with unmodified bitumen. Similar trend was observed at 64 ℃ and 70 °C for both 0.5 wt% and 1 wt% CNTs. It can be inferred that 0.5 wt% and 1 wt% of CNTs upgraded the bitumen to 70 ℃ grade. Since the increments in the rut parameter of bitumen modified with 0.5 wt% and 1 wt% are very close, 0.5 wt% of CNTs is recommended as candidate percentage for bitumen modification to improve the rutting performance of asphalt concrete.

3.3. *Marshall stability and flow of asphalt mixture*

The Marshall stability and flow of unmodified and CNTs-modified asphalt concrete are presented in Fig. 7a & b. The Marshall stability was evidently improved by adding CNTs into asphalt concrete. ANOVA was also performed on the Marshall test measurements and the results are presented in Table 4. The p-values in Table 4 are less than 0.002 revealing a significant effect of CNTs on both Marshall stability and flow. The graph showed that maximum Marshall stability was observed at 0.5 wt% CNTs recording 11.4% increase in comparison with unmodified sample. Adding CNTs of more than 0.5 wt% did not change the increment in Marshall stability; in fact, it dropped by 1% at 1.0 wt% compared to 0.5 wt% CNTs-modified sample. This drop can be considered within the experiment error. It is recommended that content of CNTs to modify bitumen for maximum Marshall stability be 0.5 wt%. In similar fashion, modifying asphalt by CNTs decreased the flow of asphalt concrete in comparison with the unmodified mix. At 0.5 wt%, the flow of asphalt decreased by 14.2%. These changes in asphalt concrete upon modification by CNTs are attributed to physical and mechanical properties of CNTs: (i) CNTs have high aspect ratio with superb mechanical strength and Young's modulus thus they can effectively transfer stress and delay crack propagation; (ii) the nanosize of CNTs makes them ideal filler to fill the pores and voids within the asphalt concrete providing denser structure in comparison with the unmodified asphalt mix [50]. Therefore, CNTs-modified asphalt concrete has high stability and resistance to flow. However, at high fractions such as 1.0 wt%, CNTs agglomerate and do not uniformly disperse within the asphalt due to the high van der Waal force. In composites, filler agglomeration diminishes the reinforcement effect [51]. Similar results were reported by previous studies [52,53] where enhancements in the Marshall stability was observed upon adding CNTs to asphalt concrete. However, these studies did not show the dynamic deformation (rutting) of the modified asphalt concrete.

The results of Marshall stability and flow measurements recommended an optimum content of 0.5 wt% CNTs to modify the asphalt with maximum stability and acceptable level of flow. Therefore, 0.5 wt% CNTs-modified asphalt concrete was selected for wheel tracking test.

3.4. *Low temperature cracking test*

Low temperature weather would make asphalt pavements more brittle in comparison with those at room temperature. It is noteworthy that brittle asphalt concrete does not withstand dynamic loads and prone to high density of microcracks. This will accelerate crack propagation within the asphalt concrete letting water ingress into the bottom layers of the pavement; this would lead to high maintenance cost and short service life. The current study aims to investigate the effect of CNTs on reducing crack density and/or delaying its propagation in asphalt mixtures at low temperature. The low cracking test was started from + 20 ℃ with a cooling rate of 10 ℃/hr till failure. The failure temperature ($T_{failure}$) and cryogenic stress (σ_{cry}) of the tested samples (0–1.0 wt%) were recorded in Table 5. Moreover, the measurements were statistically analysed using ANOVA test and its results are provided in Table 6. Boxplots and residual analysis of ANOVA are presented in Figs. S11–S14 in the supporting information. The ANOVA shows that CNTs have an

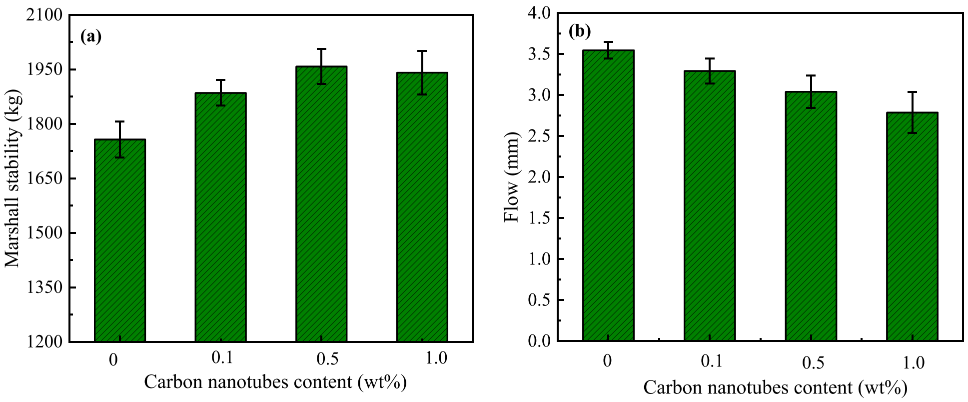

Fig. 7. (a) Marshall stability and (b) flow of unmodified and CNTs-modified asphalt mixtures.

effect on promoting the cryogenic strength of asphalt mixture at low temperature; the p-values are less than 0.04. Fig. 8 shows the results of the thermal induced stress test of unmodified and 0.5 wt% CNTs-modified asphalt mixtures. CNTs at 0.5 wt% decreased the failure temperature of the asphalt mixtures from − 24.8 ℃ for unmodified asphalt mixture to − 26.4 ℃ along with an increase in cryogenic failure stress from 4.3 to 4.55 MPa, respectively. Also, the cryogenic stress at − 20 ℃ was decreased from 2.75 MPa for unmodified asphalt mix to 2.55 MPa when the asphalt mix is modified by 0.5 wt% CNTs. When CNTs' content was increased from 0.5 wt% to 1.0 wt%, marginal effect on the cryogenic strength was observed.

Due to the high aspect ratio of CNTs, they could work as crack inhibitors and improve failure stress; CNTs can work as nano-bridges that might connect edges of microcracks. Also, being at nanosize, CNTs can fill pores and voids inside the asphalt concrete resulting in denser hot mix asphalt. Both advantages would help the asphalt mix to resist crack propagation and have longer service life in comparison with unmodified asphalt. However, we believe adding CNTs alone cannot improve cryogenic failure strength. Therefore, further research and investigation are needed to select other modifiers that can synergistically work with CNTs at low temperature for high resistance to crack propagation without compromising the performance at high temperature (rutting resistance).

3.5. *Indirect tensile strength*

The indirect tensile strength of unmodified hot asphalt mix and CNTs-modified hot asphalt mix is presented in Fig. 9. The statistical analysis by ANOVA is presented in Table 7. It is evident that modifying bitumen by CNTs enhanced the indirect tensile strength of the asphalt concrete; upon modification by 0.5 wt% of CNTs, the indirect tensile strength was elevated by 38%. Tensile strength is crucial to obtain high asphalt pavement resistance to cracks, which means better pavement performance and longer life span. A major part of tensile stress generated in asphalt mixture sample is endured by asphalt, which binds aggregates together. Therefore, adding CNTs into asphalt could strengthen the binding between asphalt and aggregates leading to asphalt concrete with high mechanical performance. The indirect tensile strength did not have a pronounced increment at 1 wt% CNTs in comparison with 0.5 wt%. This was due the fact that CNTs have high chance to agglomerate at high dosages; similar observations were noticed in the rheological measurements.

3.6. *Dynamic stability and rut depth*

Wheel tracking test is one of the most important tests to assess the deformation of hot mix asphalt under dynamic loading. Rut depth – after number of passes with a loaded wheel – evaluates the capacity of asphalt mix to withstand the traffic loads and its life span; the smaller the rut depth, the better the dynamic stability. It is noteworthy that the rheological properties of CNTs-modified bitumen, and indirect tensile strength and low temperature-cracking tests for the asphalt mixtures showed that 0.5 wt% was the optimum content for modification. Therefore, in this study, wheel tracking test was performed on two slabs of asphalt concrete: (i) unmodified asphalt concrete and (ii) 0.5 wt% CNTs-modified asphalt concrete. Fig. 10a & b presents results of the test using a 70 kg-loaded wheel at 60 ℃. ANOVA results in Table 8 shows that adding CNTs into hot asphalt mixtures significantly enhanced the asphalt mix resistance to dynamic deformation in comparison with unmodified asphalt mix. For example, after 5000 cycles the rut depth was reduced from 6.9 mm for unmodified sample to 3.8 mm upon adding 0.5 wt% CNTs representing 45.5% improvement. After 10,000 cycles, the rut depth of modified was only 4.46 mm in comparison with 8.12 mm for the unmodified sample.

Adding CNTs increased the mechanical resistance of asphalt mix to deformation due to the excellent strength of CNTs and their exceptional aspect ratio. Both features can delay crack propagation and gives better dynamic stability over the life span of pavement resulting in reduced maintenance costs. This would solve rutting distress on highways in Egypt due to hot climate and heavy traffic loads.

3.7. *Cost analysis*

In general, the expensive cost of nanoadditives limits their application in asphalt industry [54–56]. Therefore, modifying asphalt mixtures by nanofiller such as CNTs must be economically justifiable. This section studies the initial cost of modifying asphalt mixture by CNTs additives. The cost analysis was made for constructing a section of a 1-km length highway with 6 lanes (3 lanes in each direction). The specific gravity of asphalt mixture was considered as 2.3 ton/m^3. We assumed the price of one ton of asphalt is US$ 60 and that of CNTs as US$ 130/kg [57]. The costs of preparing the proposed highway with unmodified and modified asphalt mixtures were calculated using the following equations [54–56]:

(i) Unmodified asphalt mixture:

$$Total \quad cost = 1000 \times 6 \times 3.65 \times \frac{D_o \times 2.54}{100} \times \gamma \times \text{asphalt price} \tag{1}$$

(ii) Modified asphalt mixture:

$$Total \quad cost = cost \quad of \quad asphlat \quad layer + cost \quad of \quad CNTs \quad added \quad to \quad the \quad mixture$$

$$= \quad 1000 \times 6 \times 3.65 \times \frac{D_i \times 2.54}{100} \times \gamma \times \text{asphalt price} + \quad 1000 \times 6 \times 3.65 \times \frac{\mathrm{D_i} \times 2.54}{100} \times \gamma \times 1000 \times \frac{5.2}{100} \times \text{wt\%} \times \text{CNTs}'\text{price} \tag{2}$$

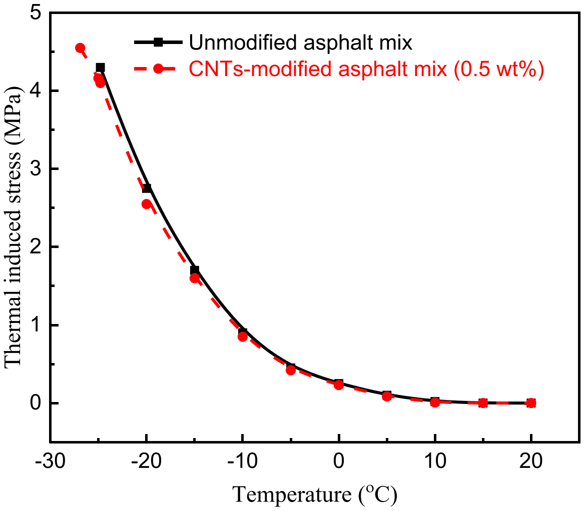

Fig. 8. Low temperature cracking test results of unmodified and CNTs-modified asphalt mixtures.

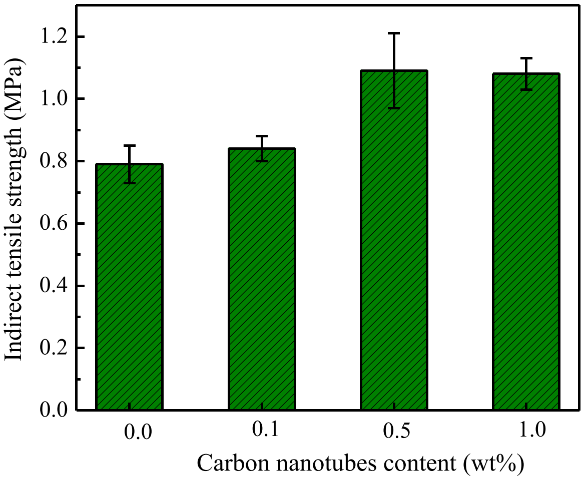

Fig. 9. Indirect tensile strength of unmodified and CNTs-modified hot asphalt mix.

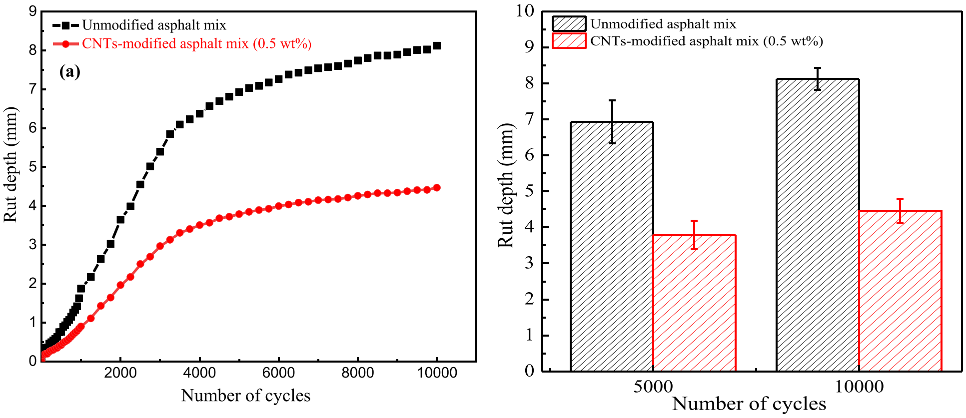

Fig. 10. Wheel tracking test results of unmodified and CNTs-modified asphalt mixes (a) rut depth over 10,000 cycles and (b) reduction in rut depth after 5000 and 10,000 cycles.

where ɣ is the specific gravity of asphalt mixture and D_o and D_i are the thickness of asphalt layer for unmodified and modified asphalt mixtures, respectively. Thickness of asphalt layer is determined using equation $D_i = \frac{SN_i}{a_1}$ where SN_i and a_1 are respectively the structural number and layer coefficient. The stability of unmodified asphalt mixture is 1756 kg and the layer coefficient at this stability is at the maximum value of 0.54 with a layer thickness of 3.75 in. Since CNTs increased the stability of asphalt mixture, the corresponding layer coefficient stayed at the maximum value of 0.54 and thus it will have the same layer thickness [54–56]. Therefore, the initial benefit due to reduction in the asphalt layer thickness is zero.

Fig. 11 shows the initial cost for unmodified asphalt mixture and CNTs-modified asphalt mixture at various contents of CNTs. It is evident that modifying asphalt mixtures by CNTs adds significant amount of extra cost for the asphalt industry. For example, the added cost of modifying asphalt by 0.5 wt% CNTs in paving the proposed highway is US\$163 $\times$ 10^3 representing 57% increase in the initial cost. However, we believe that the obtained enhancements in asphalt concrete, in particular rutting resistance, can reduce large portion of the maintenance works and prolong the pavement service life thereby offsetting the added initial cost. Also, it suggests that CNTs-modified asphalt can be used in restricted areas where there is a potential for deterioration such as turns and congested traffic lights areas.

4. Limitations and recommendations

The results of the current study confirmed the positive effect of CNTs on promoting the rutting and crack resistances of the asphalt mixtures. However, it increased the kinematic viscosity of the binder, yet it is within the permissible limits of viscosity [47]. There are limitations in the current study due to the constrains of measurement facilities and the available data. For example, we measured permanent deformation (rut depth) of asphalt mixture at only one content of CNTs due to the availability of Hamburg wheel-tracking machine. Further measurements are needed to assess the rutting resistance of asphalt mixture at various fractions of CNTs. Though the cost of adding CNTs was examined and resulted in zero-cost benefit at the initial stage, long term cost benefit of using CNTs in asphalt industry is required. Nonetheless, it was not carried out in the present study due to limited available data.

Moreover, potential and timely research directions in the area of nanofiller-modified asphalt are recommended. This includes (i) studying synergy between nanofillers and traditional modifiers, (ii) investigating chemical modification of nanofillers before adding into asphalt which can build strong bonding between nanofiller and asphalt matrix, (iii) studying other functional properties including self-healing, thermal and electrical conductivity, and wear resistance of CNTs-modified asphalt mixture; and most importantly (iv) developing a comprehensive model to investigate the economic impact of using carbon-nanomaterials in the asphalt industry. This should include not only direct cost of carbon nanomaterial but also savings and/or expenses due to the change in periodic maintenance, materials, road detouring and traffics… etc.

5. Conclusions

Mechanical properties of modified asphalt cement and mixture with carbon nanotubes (CNTs) were evaluated in this study. Three different percentages of CNTs (0.1%, 0.5%, and 1% by mass of asphalt cement) were used to modify conventional asphalt cement (60/70). The study showed that modifying asphalt binder (asphalt cement) can significantly enhance mechanical performance of the asphalt concrete in particular rutting resistance and indirect tensile strength. The following conclusions can be drawn from this study:

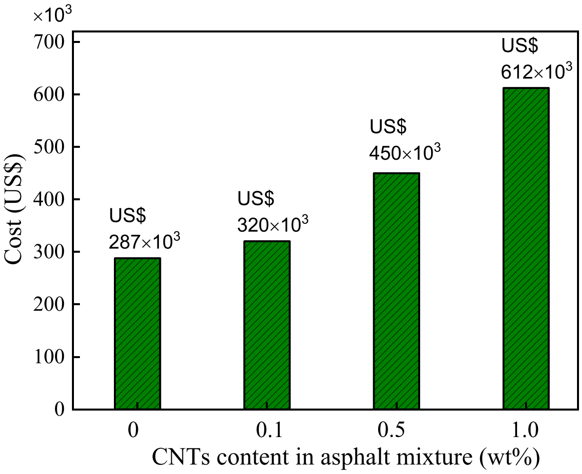

Fig. 11. Initial cost of surface course for modified and unmodified asphalt mixture.

1. The physical properties of the modified asphalt cement were enhanced upon adding CNTs. The modification of asphalt cement by CNTs decreased the penetration and increased softening point and viscosity of the asphalt cement. Addition of 1 wt% of CNTs decreased the penetration grade by 16%, and increased softening point and kinematic viscosity by 6.8% and 17%, respectively.
2. Marshall stability increased upon modifying asphalt binder by CNTs while Marshall flow decreased. The Marshall stability reached its maximum value at 0.5 wt% CNTs at flow of ~3 mm.
3. Rutting parameter increased with CNTs at the given temperature for both unaged and RTFOT-aged samples.
4. Wheel tracking test was performed on unmodified and 0.5 wt% CNTs-modified asphalt concretes. The findings were outstanding; the rut depth was reduced by 45% for the modified sample in comparison with the unmodified one. This result was consistent with penetration grade and softening point measurements.
5. The performance of modified asphalt mixture was improved at low temperature by adding CNTs; the asphalt mixture became more resistant to low temperature cracking, where 0.5% CNTs decreased failure temperature from − 24.8 to − 26.4 ℃ and increased its fracture strength from 4.3 to 4.55 MPa.
6. The CNTs modified asphalt mixture shows better indirect tensile strength values compared with unmodified asphalt mixture.
7. Modifying asphalt cement with CNTs increases the mechanical properties of asphalt concrete and CNTs-modified asphalt concrete has the potential to resist rutting, fatigue and crack propagation.
8. Even the modifying asphalt mixture by CNTs (0.5 wt%) increased the initial cost of asphalt mixture by 57%, the enhancements in asphalt concrete with CNTs can reduce large portions of the maintenance work and prolong the pavement's service life.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.cscm.2022.e00930.

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