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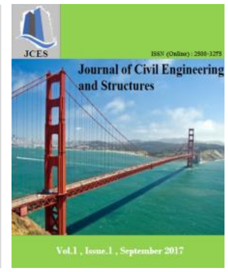

Experimental study of Nano clay and crumb rubber influences on mechanical properties of HMA

Mohsen Zahedi [1], Babak Baharvand [2*]

[1]Assistant Professor, Department of Civil Engineering, Razi University, Kermanshah, Iran.; Zahedi@razi.ac.ir
[2]Highway and Transportation Department, Islamic Azad University of Zanjan, Zanjan, Iran.; babak.baharvand65@gmail.com
[*] Correspondence: babak.baharvand65@gmail.com; Tel.: +98-916-369-2762

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Abstract: During the recent years, increased traffic load, axis load and tire pressure have influenced asphalt mixtures functions and has decreased transportation and shortened their lifetime. Although asphalt mixture function depends up on its material properties, precise design and manufacturing quality, using Nano materials and crumb rubber in asphalt mixture is improving for asphalt function enhancements. The study purpose is to investigate the impact of using Nano clay and crumb rubber together on mechanical properties of binder layer asphalt mixtures by Marshall and conventional bitumen tests by modifying the bitumen properties by the crumb rubber and substitution of usual fillers by Nano clay. In this study the crumb rubber (5, 10, 15, 20 and 25 weight percent of bitumen) was mixed with the pure bitumen 60/70 in wet method and the Nano clay was used with 5 different weight percent (from 1 to 5 weight percent of the aggregates). The study results showed that Marshall strength increased by increasing Nano clay and crumb rubber composition in the mix, and the highest strength obtained with 10% crumb rubber and 5% Nano clay portions. In addition, by increasing Nano clay-crumb rubber portion in the asphalt mixture, compared to the control sample, the aggregate pore volume increased, the total pore volume of the whole mixture was relatively increased and the mixture special gravity was slightly decreased. Also, by doing the bitumen tests it was observed that the modified bitumen thermal sensitivity decreased and its viscosity and penetration degree increased by increasing the crumb rubber portion.

Keywords: Nano clay, Crumb rubber, Asphalt mixture, Binder, Marshall test, Thermal sensitivity, HMA.

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1. Introduction

Early failures on pavements has led to less utilization lifetime of the pavements and the road users' safety and also increased maintenance costs, travel time and the users' expenses. Topeka and Binder, as the upper layers in asphalt paving on which the highest stresses of vehicle transports are implied are the most important layers. Therefore, many researches have been conducted on the improvement of these layers function in asphaltic mixtures. Today, modification methods and modern additives are used for paving improvement. In this respect, this study purpose is to evaluate the influence of adding both Nano clay and crumb rubber on improvement of mechanical properties of the asphaltic mixtures, based on the experimental investigations.

Nano technology is in fact the ability to produce new materials, tools and systems in molecular or atomic scale which affect the material interactions in microscopic scale [28].One Nano structure material has been widely used to modify asphaltic mixture properties is Nano clay. Nano clay plays a filling role in

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asphaltic mixtures and can improve mechanical properties of asphaltic mixtures by increasing adhesion between bitumen and aggregates [24].noted in a study that using Nano Clay increased asphalt long time efficiency and adding 2% Nano clay to the bitumen, led to increased number of passed cycles to reach the second and third phases of bending failure [10]. In addition, noted in his M.S paper that increased Nano clay up to 3% in asphaltic mixtures showed a higher strength [7].Nano clay improves asphaltic mixture function.concluded in a study that using Nano clay in asphaltic mixtures led to longer pavement lifetime [5].

Despite its low weight percent (4-6%), bitumen plays an important role in strength and stability of the road pavement against weathering factors and therefore any improvement in bitumen properties leads finally to the pavement function improvement [23]. However, if it is provided with the highest quality, bitumen is a fluid with certain properties and limit functions and has no significant elastic properties. Thereby, in many cases, it is necessary to improve this feature by adding new modifier materials. Despite of its widely uses in asphaltic pavement, bitumen has the problem of low brittleness temperatures and fluidity at high temperatures. This phenomenon is called "thermal sensitivity" [2]. In the recent years, many efforts have been made to modify physical and rheological properties of bitumen by using different additives. One of these additives is polymers. Addition of polymer modifiers (OL, EL, and SB) into the bitumen has positive influences on properties and its function at high and low temperatures, the permanent deformation resistance and thermal cracks [18]. Polymers decrease bitumen thermal sensitivity, especially at higher temperatures in which bitumen flows [12]. Polymers used to modify bitumen categorized into two groups: one is plastomeric modifiers e.g. poly propylene and poly ethylene, and the other is elastomeric modifiers e.g. Styrene Butadiene Styrene (SBS), natural rubber and crumb rubber. Elastomeric polymers especially crumb rubber are more concerned for their availability and low cost and also environment friend benefits than other kinds of polymers [12,20].

Research shows that slightly addition of rubber into the bitumen makes significant shifts in physical properties of it and if they are compared, it will be seen that the modified bitumen with rubber owns some properties like lower thermal sensitivity, more strength against deformation and high temperatures, and lower brittleness at lower temperatures [25].note in their paper that adding rubber into the bitumen at higher temperatures improves strength against permanent deformation or rotting [16].in their paper show that increased crumb Rubber percent decreased the penetration degree, thermal sensitivity, ductility and Fraass breaking point and increased softening point, elastic recovery and Vialit adhesion [15].In addition to the positive effects of crumb rubber on bitumen properties,concluded in their paper that addition of crumb rubber into the asphaltic mixture, regardless of type and size of the crumb rubber, improved strength against rotting in asphaltic mixtures and also increased fatigue time and VMA [26].in another study showed that Superpave mixed design method and volumetric analysis could be used in asphaltic mixtures modified with crumb rubber. They also noted that addition of crumb rubber into the bitumen increased optimum bitumen amount [27].Adding crumb rubber into asphaltic mixtures improves the stregth of the mixtures against rotting at high temperatures and elasticity at lower temperatures, in addition to increased bitumen viscosity which improved stregth against penetration [11].in their research showed that by using modified bitumen by crumb rubber in asphaltic mixtures, noises from interaction between the tire and pavement significantly decreased [19]. In addition, using crumb rubber in road pavement decreased demand on natural resources consumption for road construction and solved the problem of disposal of worn tires [9]. Even there exists purposive long term plans to apply rubber for road pavements in some countries, e.g. Spain where a national plan introduced to encourage using the recycled worn tires materials. The plan has introduced to initiate effective actions by the related organizations in order to guarantee application of 45% rubber in bitumen mixtures up to 2015 [19].

Regarding to the researches and previous results to distinct use of the two additives for asphaltic mixes, this study aims to evaluate the influence of using both Nano clay and crumb rubber in asphaltic mixtures with continuous grading by Marshall test (ASTM-D1559) and the conventional tests for bitumen. Therefore, this study tries to show the improvement of physical and strength properties of asphaltic mixtures by using both materials. In this study, crumb rubber (5, 10, 15, 20 and 25 weigh percent bitumen) was mixed with pure bitumen. In addition to the application of crumb rubber for asphaltic mixtures to improve the bitumen properties, their environmental benefits have also been concerned. On the other hand, Nano clay (1, 2, 3, 4 and 5 weight percent of aggregates) with properties like high mechanical strength and its filling effect, substituted fillers of aggregates. It should be highlighted that in this study, increased crumb rubber and Nano clay in asphaltic mixtures, resulted in decreased pure bitumen and conventional fillers as well. Also, results of technical specifications of optimum mixtures of the study compared to the limits of the specifications of hot asphaltic mixtures with continuous grading recommended by Asphalt Institution (1984).

2. Materials and Methods

2.1 Aggregates and Bitumen

In order to design the study mix, based on binder layer grading, aggregate materials is broken type and prepared from Asphalt company Abadrahan Pars. The aggregate material resource was mountaineer mines around Lorestan, Iran. Continuous grading curve of the asphaltic mix of the study, regarding to the upper and lower limits of the continuous grading of binder layer, recommended by Asphalt Institute (1948) is shown in figure1. Aggregate materials composition percent of the binder layer is shown in table1. In addition, the applied bitumen in the study is pure bitumen 60/70 prepared from Isfahan refinery, Iran. Physical and rheological properties of the bitumen used in the study are shown in table 2.

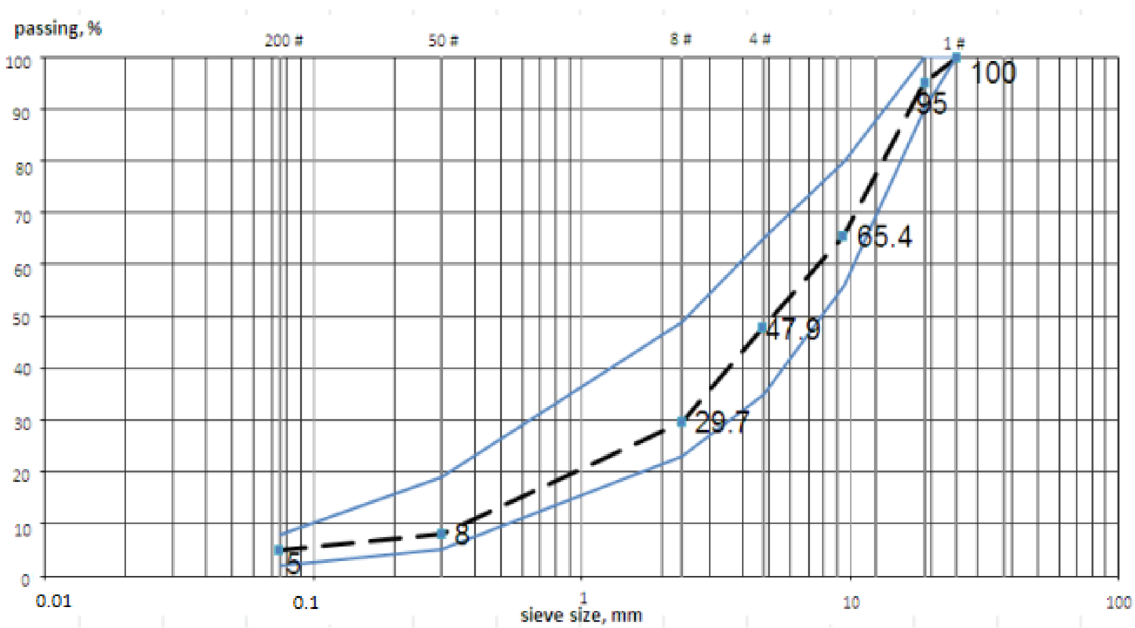


Figure 1. Aggregate materials grading of binder layer

Table 1. Ratios of aggregate mixing of the asphaltic mix

Aggregate properties	Mix percentage
Gravel (19-25 mm)	12
Gravel (12-19 mm)	23
Gravel (6-12 mm)	17
Sand (0-6 mm)	43
Filler	5

Table 2. Specifications of the used bitumen

Property	Range	Test method	Result
Specific gravity at 25 °C	-	ASTM-D70	1.016
Penetration at 25 °C (in 0.1 mm)	60-70	ASTM-D5	64
Softening point (°C)	49-56	ASTM-D36	49
Kinematic viscosity at 25 °C (cSt)	-	ASTM-D2170	357
Ductility at 25 °C (cm)	100	ASTM-D113	106
Flash point (°C)	Min. 232	ASTM-D92	310
Solubility in trichloroethylene (%)	Min. 99	ASTM-D2042	99.11
Drop in penetration after heating (wt%)	Max. 0.8	ASTM-D6	0.01
PI (penetration index, in terms of softening point and penetration at 25 °C)	-	-	-0.8677
PVN (25-135)	-	-	-0.8695

2.2 Nano clay

Although material properties is important for engineers in Meso and Macro scales, Micro and Nano scales make noticeable advances in science and technology development (figure 2) [28]. Nano clays are clay minerals, at least one of its dimensions is in Nanometer and mostly used as filler to modify polymer properties. Purity and cationic exchange capacity are two important specifications for Nano clay success in polymers strength. Nano clay is created by separation of the clay plates which has a special big active area between 700-800 m2 per each gram. This big active area makes a strong interaction between Nano clay and surrounding media (e.g. bitumen) [6].

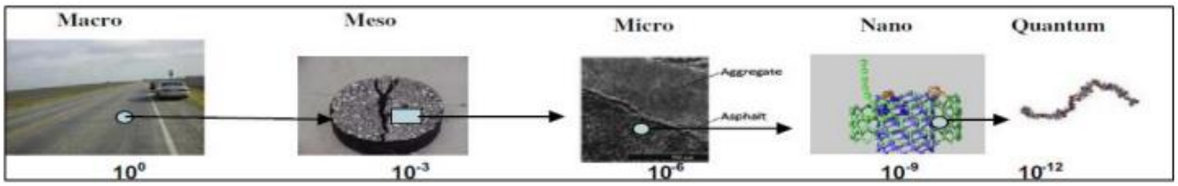


Figure 2. Description of different asphaltic scales [28]

One other Nano clay specification is the strength against heat, prevention of gas penetration and mechanical strength [17]. In this study Montmorillonite Nano clay (figure 3) was used, prepared from Nano sunny Co. products of Rockwood additives Co., specifications of consumed Nano clay of the study is presented in table 3.

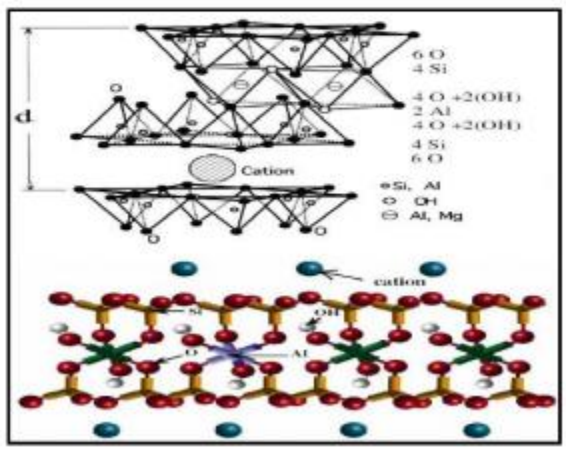


Figure 3. Montmorillonite structure [6]

Table 3. Nano clay properties [21]

Property	Value(s)
Type of mineral	Montmorillonite (Na^+)
Specific gravity (gr/cm^3)	5-7
Moisture content (%)	1-2
Specific surface area (m^2/gr)	500-750
Electrical conductivity (MV)	25
Ion exchange conductivity (meg/100 gr)	48
Void distance between particles (Aº)	60
PH	7.30-7.60
Particle size (nm)	1-2

2.3 Crumb rubber

Crumb rubber has been used as an additive in asphaltic mixtures [1]. There have been introduced three sources of rubber for bitumen modification yet: natural rubber (Latex), crumb rubber (tires rubbers) and artificial rubber (Styrene–Butadiene). The obvious specification of rubber is its high elasticity such that on load conditions, it can tolerate a high deformation and returns to its original status right after unloading. This feature relates to the molecular structure of the rubber. Rubber is a polymer and in fact an elastomer which has long molecules connected in different points chemically or mechanically and create a three dimensional network [25]. Althogh rubber as well as bitumen, is a thermoplastic or viscoelastic material and its deformation depends on temperature and strain amount under loading, its elastic deformation is less sensitive against thermal change and preserves its elastic properties under low strain and high temperatures. Wide range of rubber elasticity behavior compared to bitumen is resulted from cross link bonds and long molecules. Also, its stretch feature is more than that of bitumen at lower temperatures and heavier loading conditions [25]. In general, there are two methods to use crumb rubber in asphaltic mixtures, one is wet process with particle size (0.6- 0.15 mm) and the other is dry process with particle size (0.85-6.4 mm), any approach in which crumb rubber and bitumen mixed before adding bitumen into the aggregates is a wet process. In a dry process crumb rubber is directly added into the hot asphaltic mixtures (aggregates) [9,13].

In this study, wet process was used to mix the crumb rubber and bitumen. Crumb rubber particles used in this study were smaller than Sieve mesh No.40 and its grading curve is seen in figure 4, which was widely used in production of modified crumb rubber mixtures (CRM) in south Carolina in the U.S. [8]. The crumb rubber used for this study produced by Azar Sam Qazvin Co. Iran shown in table 4.

3.Experiments

3.1 Marshall test (ASTM-D1559)

In order to make asphaltic mix using Nano clay and crumb rubber, the initial mixing was used (according to table 1). With a difference that in this phase pure bitumen was modified by different composition percent of the crumb rubber and the filler was substituted by different percent of Nano clay. To do this, crumb rubber and pure bitumen 60/70 were mixed in wet process at 150 – 155 ° C. Mixing time was 15 min and mixing rate was selected 700 rpm. It should be noted that in mixing crumb rubber and pure bitumen, increasing weight and percent of the crumb rubber decreased the weight and percent of the bitumen, consequently.

Samples were compressed at 135 °C and applying 75 impacts to each side. The samples were cooled one day in ambient temperature and were immersed into 60 °C water for 30-40 minutes before loading in a 51 mm/min rate using Marshall Jack.

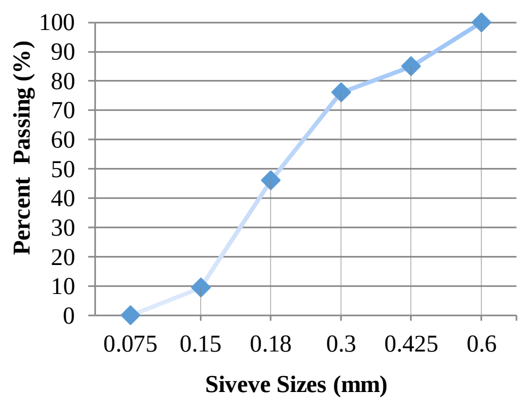


Figure 4. grading curve of the crumb rubber

Table 4. Crumb rubber properties

Property	Value
Specific gravity (gr/cm^3)	1.16
Ash content (%)	5
Plastic content (%)	10
Black carbon content (%)	29
Polymer content (%)	50
Particle size (mm)	0.0-0.42

3.2 Bitumen tests

Before making asphalt samples, bitumen 60/70 was heated up to 150 -155°C in the lab and then each crumb rubber percent (5, 10, 15, 20, and 25 weight percent in bitumen) added into the bitumen by a salt shaker and mixed for 15 min in a blender with 700 rpm rate. There are many factors which play vital role in interaction between bitumen and crumb rubber particles. Since this interaction highly depends up on the type of aromatic oils in bitumen, the bitumen resource significantly influences the bitumen features modified by crumb rubber [15]. To do this, penetration degree (ASTM-D5), softness point (ASTM-D36) and viscosity (ASTM-D2174) tests were performed. Also, in order to evaluate the influence of different portions of the crumb rubber on penetration index and thermal sensitivity of the modified bitumen, equations (1) and (2), suggested by Huang, were used [3]. Viscosity number of penetration and parameters L and M were calculated by equations (3), (4) and (5) [14], where T is the penetration test temperature and T_{RB} is the softening degree:

$$(\mathrm{PI}) = \left[\frac{20 - 500\mathrm{A}}{1 + 50\mathrm{A}}\right] \quad (1)$$

$$\mathrm{A} = \left[\frac{\mathrm{LogT} - \mathrm{Log800}}{\mathrm{T} - \mathrm{T_{RB}}}\right] \quad (2)$$

$$\mathrm{PVN} = \frac{-1.5 * [\mathrm{L} - \mathrm{Log(Viscosity135°C)}]}{\mathrm{L} - \mathrm{M}} \quad (3)$$

$$\mathrm{L} = 4.25800 - 0.7967 \times \log(\mathrm{T}) \quad (4)$$

$$\mathrm{M} = 3.46289 - 0.61094 \times \log(\mathrm{T}) \quad (5)$$

4.Results

4.1 Marshall strength

Marshall strength is a function of type, quality and specification of the filler and the modifier. Nano clay fills the pores between aggragates, with its high mechanical strength and filling effect. Thereby, Nano clay particles can cause better link between the larger aggregates in asphaltic mix and a compact structure of the aggregates. Therefore, the more Nano clay portion, the more the mixture stregth. Crumb rubber also enhances the compact structure of the Nano clay particles and aggregates by influencing the pure bitumen and lasting the asphaltone phase of the modified bitumen and creating a bitumen with lower viscosity. Consequently, addition of 10% crumb rubber could increase Marshall strength up to 40% against the control samples. Since the bitumen weight decreased by increasing the crumb rubber portion into it, the curve ascending trend did not continue by increasing the crumb rubber portion more than 10 weight percent (figure 5).

4.2 Voids in mineral aggregates (VMA)

In asphaltic mix desing, minimum volume of pores in aggregates depends up on the largest nominal size of the aggregates which increases by finer aggregates. Nano caly particles result in thickening of bitumen layer around the aggregates. Since crumb rubber particles do not ressolve in bitumen, it may thicken the bitumen layer around the aggregates. Consequently, as it can be seen in figure 6, the pore volume of aggregates shows 9% increase by rising the portion of Nano caly and crumb rubber in the asphaltic mixture.

4.3 Flow

Marshall flow is a function of type, quality and specifications of fillers. As nano clay particles improve adhesion between aggregates and bitumen, flow decreaes 67% against the control sample in the asphaltic mixture by increasing Nano clay and crumb rubber portions (figure 7).

4.4 Voids in total mixture (VTM)

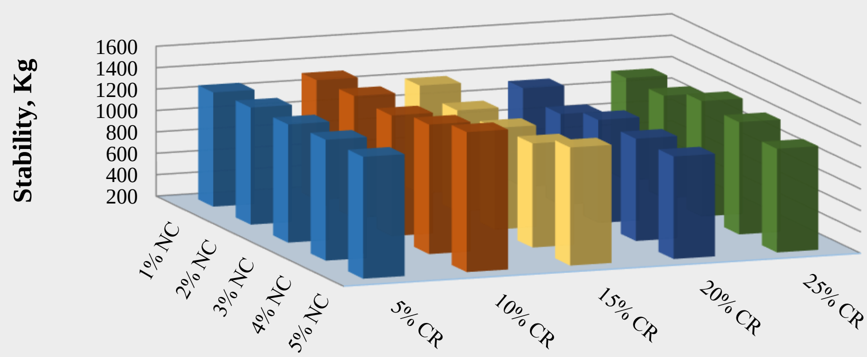
Total mixture pore has increased about 36% against the control sample by increasing crumb rubber and Nano clay portions in the mixture which could be a result of chemical interaction between Nano clay and crumb rubber in the asphaltic mixture (figure 8).

4.5 Voids filled with asphalt (VFA)

According to figure 9, it can be seen that the volume of the filled pores with bitumen has decreased up to 8% agianst the control sample by increasing Nano clay and crumb rubber portions.

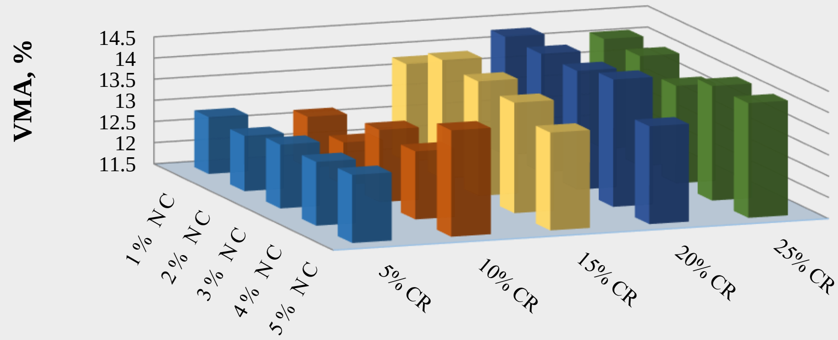
4.6 Special gravity

Special gravity has decreased 2% against control samples by increasing Nano clay and crumb rubber portions in asphaltic mixtures (figure 10).



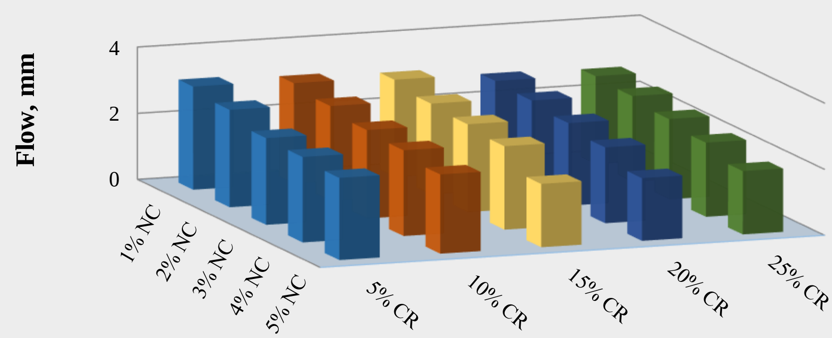
	1% NC	2% NC	3% NC	4% NC	5% NC
5% CR	1267	1293	1303	1327	1336
10% CR	1321	1340	1326	1408	1505
15% CR	1209	1145	1148	1171	1301
20% CR	1124	1050	1162	1154	1156
25% CR	1158	1159	1277	1248	1167

Figure 5. Results of Marshall strength against different portions of crumb rubber and Nano clay



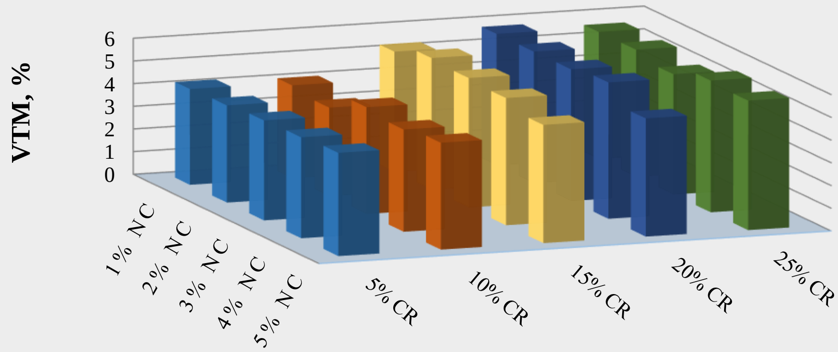
	1% NC	2% NC	3% NC	4% NC	5% NC
5% CR	12.84	12.8	13	13	13.1
10% CR	12.7	12.5	13.2	13.1	14
15% CR	13.8	14.3	14.2	14.1	13.8
20% CR	14.3	14.3	14.3	14.5	13.8
25% CR	14.1	14.1	13.8	14.2	14.2

Figure 6. VMA results against difffernt portions of crumb rubber and Nano clay



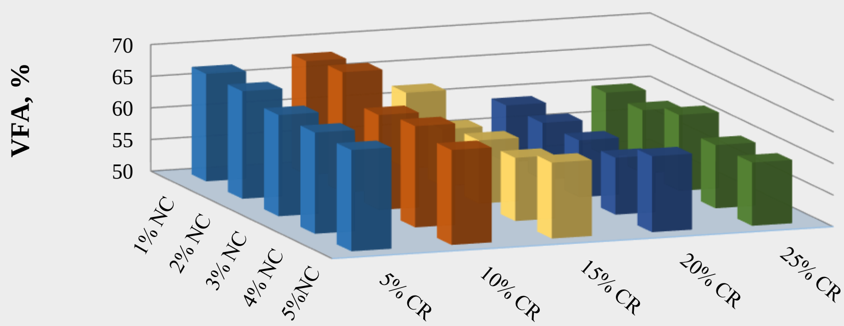
	1% NC	2% NC	3% NC	4% NC	5% NC
5% CR	3.12	2.95	2.63	2.59	2.49
10% CR	3.03	2.87	2.68	2.6	2.4
15% CR	2.94	2.75	2.66	2.53	1.92
20% CR	2.71	2.65	2.5	2.3	1.9
25% CR	2.66	2.59	2.43	2.25	1.92

Figure 7. Marshall flow test results against different portions of rubber and Nano clay



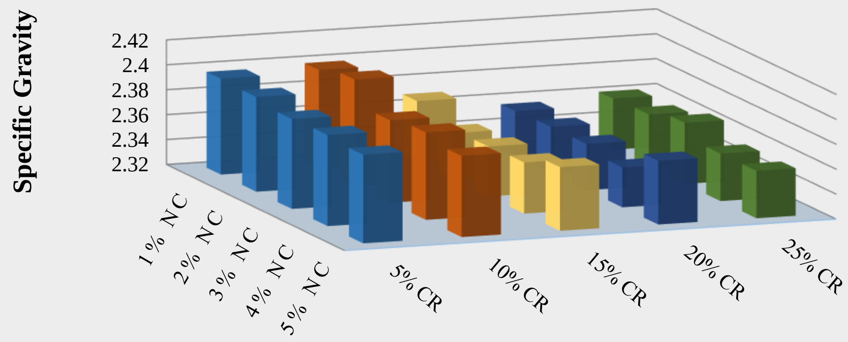
	1% NC	2% NC	3% NC	4% NC	5% NC
5% CR	4.22	4.28	4.4	4.44	4.54
10% CR	4.1	3.9	4.7	4.5	4.7
15% CR	5.3	5.8	5.7	5.6	5.2
20% CR	5.8	5.8	5.8	6	5.2
25% CR	5.6	5.6	5.3	5.8	5.7

Figure 8. VTM results against different portions of crumb rubber and Nano clay



	1% NC	2% NC	3% NC	4% NC	5%NC
5% CR	67	67	66	66	66
10% CR	68	69	65	66	65
15% CR	62	59	60	60	62
20% CR	59	59	59	59	62
25% CR	60	60	62	60	60

Figure 9. Results of VFA test against different portions of crumb rubber and Nano clay



	1% NC	2% NC	3% NC	4% NC	5% NC
5% CR	2.397	2.396	2.392	2.393	2.391
10% CR	2.399	2.405	2.386	2.39	2.385
15% CR	2.369	2.357	2.36	2.361	2.371
20% CR	2.356	2.357	2.357	2.352	2.371
25% CR	2.361	2.362	2.369	2.358	2.358

Figure 10. Results of special gravity against different portions of crumb rubber and Nano clay

5.Results of bitumen tests

As in this study crumb rubber was mixed with the pure bitumen 60/70 in 5, 10, 15, 20 and 25 weight percent, and used to prepare Marshall test samples along with Nano clay, determination ofphysical properties of the bitumen modified with crumb rubber seems to be vital. In this regard, degree of penetration (ASTM-D5), softening point (ASTM-D36) and viscosity (ASTM-D2174) tests were conducted and the results and also penetration degree parameter and thermal sensitivity on the modified bitumen are presented in table 5. Finally, regarding to the limits of physical and strength characteristics of hot asphaltic mixtures by Marshall test, suggested by asphalt institute, according to table 6. In conclusion, results from the study and limits determined by asphalt institute are compared on physical and strength properties of the asphaltic mixtures.

Table 5. Results of the bitumen test modified with different portions of crumb rubber

Crumb rubber	Degree of penetration (0.1 mm)	Softening point (°C)	Viscosity (centi stocks)	Thermal sensitivity	Penetration index
blank	64	49	357	0.04570	-0.8676
5	64	50	465	0.04388	-0.6074
10	62.5	53.5	985	0.03885	+0.1954
15	59	57.5	1852	0.03484	+0.9409
20	53.2	61	5391	0.03269	+1.3874
25	47.5	64.8	11920	0.03081	+1.8087

Table 6. Physical and strength properties of hot asphaltic mixtures by Marshall test (Asphalt Institute 1984)

description	Heavy traffic EAL ≥ 10^7		Average traffic 10^4 < EAL < 10^7		Light traffic EAL < 10^4	
	min	max	min	max	min	max
Number of impacts both sides of the sample	75	75	50	50	35	35
Mixture strength (kg)	800	-	550	-	350	-
Flow (mm)	2	3.5	2	4	2	4.5
Voids of asphalt binder (%)	3	5	3	5	3	5
Voids filled with asphalt (%)	65	75	65	78	70	80
Min voids between aggregates (VMA)	According to table 5-03 of the same reference					

6.Conclusion

During the recent years, researchers and engineers have tried to modify properties and function of asphaltic mixtures by using modern approaches. The recent years researches show that Nano materials and crumb rubber significantly influence the modification of asphaltic mixture properties. Therefore, in this study, different portions of both Nano materials and crumb rubber were evaluated. Crumb rubber in the asphaltic mixture (5%, 10%, 15%, 20% and 25%), as an elastomeric polymer with high elasticity and lower sensitivity could improve physical and rheological properties of pure bitumen against temperature changes. On the other hand Nano clay (1%, 2%, 3%, 4% and 5%) could improve physical properties and increased durability of asphaltic mixtures, with a high mechanical strength and its filling influence in the mixture.
Regarding to the test results, the total results of the study are as follow:

- Degree of penetration of the modified bitumen showed 26% decrease against the control sample (without additive) by increasing the portion of crumb rubber added into the bitumen. This decrease might relate to the saturation of the bitumen by crumb rubber when using high portions of crumb rubber and also increasing time of asphaltene phase by decreasing maltene phase by the crumb rubber which could finally lead to the increased shear strength and decreased permanent deformation of the asphaltic mixtures at middle and high temperatures.
- Softening point of the modified bitumen showed 33% increase against the control sample (without crumb rubber) by increasing portion of crumb rubber. Simultaneously, by increasing the portion of crumb rubber, adsorption of lubricant oils by the crumb rubber increased and thereby asphaltene phase time increased which hardens the bitumen and increased elastic behavior and its softening point and also increased the temperature of bitumen functionality.
- Thermal sensitivity of the modified bitumen decreased 32% against the control samples by increasing the portion of crumb rubber. Although, the less the bitumen thermal sensitivity, the better it functions at temperature changes.
- Marshall strength showed about 1.5 times increase against the control sample by increasing both portions of crumb rubber and Nano clay (10% crumb rubber and 5% Nano clay). This increase might show the filling effect of Nano clay particles between the pores of the aggregates and creating a compact system of aggregates modified by the bitumen and enhanced by crumb rubber.
- Since crumb rubber particles do not resolve in bitumen, they stick to the aggregates with the bitumen and can increase the thickness of bitumen layer around the aggregates and finally this composition might increase VMA of asphaltic mixture against the control samples.
- Increasing of both portions of Nano clay and crumb rubber in asphaltic mixture directly relates to the mixture VTM.

- The mixture VFA decreased against the control sample by increasing both portions of Nano clay and crumb rubber.
- Increasing crumb rubber portion in the mixture led to lower special gravity which could be stemmed from increased VMA.
- Environmental pollution decreased by using crumb rubber in asphaltic mixtures, which is a disposal material, 100% recycled.

Regarding to the total results of Marshall test parameters in this study and permit limits presented in table 6 which suggested by asphalt institute (1984), optimum portion of Nano clay and crumb rubber in asphaltic mixture is 5 and 10, respectively. The optimum portion results of this study compared to the allowed limits are presented in table 7.

Table 7. Physical and strength properties of asphaltic mixture for optimum portions of Nano clay and crumb rubber

parameter	Strength (kg)	Flow	Voids in total mixture (%)	Voids in mineral aggregates (%)	Voids filled with asphalt (%)	Special gravity
Study results	1505	2.4	4.7	14	65	2.385
MS-2 limits	Min 800	2-3.5	3-5	Min 14	56-75	-

Suggestions

Despite the current studies on this field, there needs more researches on using Nano clay and crumb rubber in asphaltic mixtures along with this study, for example wet sensitivity and wheel-track tests are important.

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