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Investigating the Rheological Properties of Bitumen Clay Emulsion with Different Bentonite Emulsifiers for Pavement Application

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Abstract

Cold in-place recycling using emulsion bitumen is an appropriate option for improvement and pavement of the asphalt. The researches show that the asphalt operation against cracking, flowing and impact resistance improves with addition of clay to bitumen. To take advantage of these properties in the cold asphalt, these bitumen clay emulsions can be used. In the present research, based on the researches done, first, an appropriate formulating and method was obtained for laboratory production of the clay emulsion using the response surface method (RSM). Then, from two bentonites, a clay emulsion with weight percentages of 3, 3.5, 4.5 and 5% of each bentonite was prepared by homogenizer laboratory method. After preparation process, the emulations were tested. The results show that with the fundamental changes created in network by the bentonite, the molecules of the bitumen act like a reinforcer and lead an increase in the stiffness of the bitumen. According to the statistical investigations done on the clay emulsions produced in this study, the emulsions made by 3.5% of bentonite type-2 has generally more desirable performance than the other clay emulsions.

Keywords: *Clay emulsion; Bentonite; Emulsifier; Pavement operation; Oil derivatives.*

1. Introduction

Cold recycling of the pavement asphalts using emulsion bitumen is one of the fastest, the most effective and effective and most compatible method with the environment compare with other repair, improvement or reconstruction methods of the pavement. Cold recycling (CR) using emulsion bitumen is a choice besides having the economical and environmental advantages also enables the modification and reuse process of the top layers' materials, given the development of the advanced machineries, without damaging the substrate and its resistance.

The emulsions which used in this method include slow set anionic and cationic emulsions specially emulsion with high float medium setting. Clay emulsions are a group of the bitumen emulsions which are prepared using organic emulsifiers which include very fine-grained solid materials such as black carbon, silicon powder and also some of the clay group soil such as Kaolin, juicy mica and bentonite which have intumescent property in aqueous phase (hydrating). This property is more visible for bentonite, due to its special crystal structure, which is a type of the clay soil of Montmorillonites' group. For this purpose, the bentonite has more emulsifying property than the other materials and also some specific kind of this materials were used as a cleaner before the soaps appear [1]. Given the above-mentioned discussion, and because of the existence of the big bentonite clay mine inside the country, the advantages of the bitumen-clay mixing and the relatively lower complexity in producing the clay emulsion than the other emulsion, more researches are needed to be done in this field. Therefore, the aim of this study of to investigate the effect of bentonite additive on the properties and operation of the cationic emulsion bitumen.

In this research, the bentonite which is a king of fine-grained clay with more than 85% of montmorillonite was added to bitumen as an organic emulsifier to modifies the bitumen emulsion's properties and the properties of the recycling asphalt mixture. Based on the assumptions of this study, the bitumen, due to having high silica in its mixture and also due to proper and uniform spreading within the aqueous phase of the emulsion bitumen, leads the bitumen to be more stiffed after emulsion failure, as a result, it is expected that the it takes more time for resulted bitumen to be softened and shows a better performance.

2. Literature review

Bitumen is a material derived from the crude oil which is known as a colloidal matter composed of both continues and discrete phase. Discrete phase includes asphaltenes composed of the semi-polymeric and heavy molecules of aliphatic and aromatic which are dispersed in the bitumen as discrete and homogeneous and/or a as a coil and ball with electrostatic bond in the form of cluster [2]. In clay emulsions, external or continues phase includes water and clay which a kind of emulsifier material (anionic) added to it and after adding the internal phase or bitumen, the clay emulsion is created. According to Fig. 1, the clay particles play two significant roles in the clay emulsions; first in the production and reserving emulsion as a stabilizer which prevent the joining of the bitumen's droplet and its coagulation; secondly, after emulsion breaking and drying of the bitumen layer, the it produces a skeleton inside it which strengthen it and reduces the sensitivity to temperature increase [3]. The research done by Salmon and Newcomb, 2012, on the different kinds of the emulsion bitumen (such as CSS-1, HFMS-2s and HFMS-2p), showed that the slow-setting emulsions (such as HFMS-2p) create fewer air vents in the mixture than the other emulsions [4].

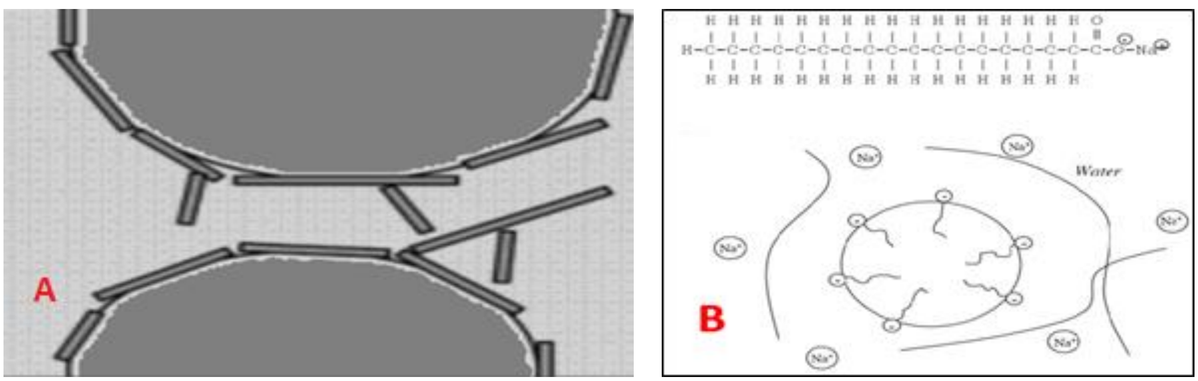

Fig. 1. A photomicrograph of a bitumen emulsion stabilized with (a) clay particles, (b) chemical emulsifier [5-6]

In a research dine by the New Mexico state transportation authority, it was found that to overcomes the rattling, reflective crack and the humidity failure problems in asphalt cold recycling, the high floating (HF) slow-setting emulsion bitumen can be used rather the SS-1 and CMS-2s bitumen [7]. Li *et al.* [8] carried out an investigation on the recycled cold asphalt mixing scheme, and found that most of the states in the USA using a kind of emulsion bitumen with high floating where most of them are of slow-setting type and few states prefer to use the slow-setting cation. Although in the cold recycling projects of the pavement, the type of the emulsion bitumen used is not completely clear and depends on the traffic, environmental conditions and the type of equipment and workshop, but generally the slow-set (SS) emulsion bitumen and medium-setting (MS) emulsion bitumen are used in full deep repair (FDR) and cold in-place recycling (CIR), respectively [9].

There are not many researches done on the application of clay in bitumen modification, but they have many applications in polymer modification. Bitumen is also a kind of polymer with very complicated chemical mixture [10], which expect that the clay has effect on its behavior. Sorkumar *et al.* [11], in 2010, investigated the effect of the additive clay in the polymer asphalt mixtures and found that the clay has a consistent effect with asphalt and polymer and the high consistency between clay and polymer can lead to better dispersion of the polymer in the asphalt, and as a result effects on the final rheological specifications of the systems under study. In a research done in 2010 by Govoh *et al.* [12], results showed that the addition of the nano-clay and micro-carbon fiber has positive effects on the indirect tensile strength of the asphalt mixtures. However, the use of natural and environmentally friendly materials in the refining of natural oils and materials derived from petroleum such as oil and bitumen has been the subject of much research, and the use of bentonite from natural sources has also been addressed [13]. According to the studies done, it is clear that there is any research done on the rheological behavior of the clay bitumen containing bentonite. Therefore, the present study addresses this topic experimentally.

3. Research method

The aim of the present study is to investigate the using of bentonite as a mineral emulsifier in bitumen emulsion production and to evaluate the effect of using this emulsion on the recycled asphalt mixture in the pavement. Therefore, based on the previous research done on the clay emulsions and the cooled mixtures recycled by emulsion bitumen and by cooperation of the chemical laboratory of the Tehran oil industry research institute, some samples of the clay emulsion were prepared and tested using two different bentonites by the experimental method to ensure the accuracy of the product produced. In order to achieve a specific formulation to produce a clay emulsion a surface response statistical method was used which is an applicable statistical and mathematical method for creating experimental method and optimizing the data of the test. In the tests performed in this research, different guidelines, standards, specifications and methodologies were used. Table 1 listed these references along with their names.

Table 1. A list of the standards and guidelines used in the present research [14]

Application	Title	Code	Standard Test
Bitumen penetration for the manufacture of clay emulsion	Standard Test Method for Penetration of Bituminous Materials	D5	ASTM
Soft bitumen point for making clay emulsion	Standard Test Method for Softening Point of Bitumen (Ring-and-Ball Apparatus)	D36	ASTM
Bitumen density for the manufacture of clay emulsion	Standard Test Method for Density of Semi-Solid Bituminous Materials (Pycnometer Method)	D70	ASTM
Ductility of pure bitumen for the manufacture of clay emulsion	Standard Test Method for Ductility of Bituminous Materials	D113	ASTM
Remaining test on emulsion sieve	Standard Test Method for Oversized Particles in Emulsified Asphalts (Sieve Test)	D6933	ASTM
Emulsion viscosity	Standard Test Method for Viscosity of Emulsified Asphalt by Saybolt Furol Viscometer	D7496	ASTM
Emulsion viscosity limit	Standard Specification for Emulsified Asphalt	M 140	*AASHTO*
Electric charge of emulsion	Standard Test Methods and Practices for Emulsified Asphalts	D244	ASTM
Emulsion stability	Standard Method of Test for Emulsified Asphalts	T59	AASHTO
Clay bitumen clay emulsion	Standard Method Of Test For Saybolt Viscosity	D88	ASTM

3.1. Bentonite

To modify the specifications of the emulsion bitumen using clay and to produce the clay emulsion, two kinds of the bentonite clay soils made be Iran were used which have high mineral storage volume and the mining companies operating these resources perform appropriate chemical and physical morphology tests on the produced products. The first bentonite extracts from the "Ghaleshor mine", Isfahan with brand name “Arya Bentonite” which hereinafter referred to as Be1 bentonite and the second one extracts from the "Langar mine Khorasan", Iran, with brand name “Farazan powder bentonite” which hereinafter referred to as Be2 bentonite. Fig. 2 shows a test image for both kinds of bentonite. The chemical analysis of bentonite samples, physical specification and their water absorption according to the relevant manufacturer's documentation as well as the results of the grading test using standard sieve series are shown in Tables 2 and 3, respectively.

As can be seen from Table 2, the average percentage of the SiO_2 in the samples is 58.87% which indicates that the silica is the main mineral in the samples. The average percentage of the Al_2O_3 is 13.59. by increasing the aluminum oxide, the physical and chemical strength of the material against pressure, tensile and heat increases. In most of soils, the adhesive percentage of water is one of the most important properties which defines the behavior of the soil and is used mostly used in volume form. In fine-grained (adhesive) soils which have the capability to absorb and maintain water, is very important in stability and strength of the soil. In water absorption test, every samples are prepared as a suspension of water-bentonite specimen mixture. After 14 hours, when the specimen reaches the saturation step, the humidity percentage of the specimen will be defined. In Table 3, the results of water absorption of bentonite specimens based on the documents provided by the manufactures have been defined.

Table 1. Grading analysis results of two types of the bentonite used

Sieve number	Bentonite Be1*	Bentonite Be2*
60	4.64	8.73
80	5.82	19.02
100	8.93	29.84
200	39.32	52.1
270	84.44	91.81
Cc	0.95	0.77
Cu	1.5	1.98
ASHTO classification system	A-7-5	A-7-5
Unified Classification System	SC	C H

**Percentage remaining on sieve of total weight*

Table 2. the chemical analysis of the bentonite used

Bentonite Be1	Bentonite Be2	Composition
5.78	2.41	Na_2O
2.81	2.93	MgO
17.60	9.59	Al_2O_3
54.60	63.14	SiO_2
2.33	3.03	CaO
1.69	2.54	Fe_2O_3
15.19	16.63	Other elements

Fig. 2. The specimens of two bentonite under the test

Table 3. Physical properties of the bentonite used

Physical properties	Bentonite Be1	Bentonite Be2
Pasty limit PL	35.5	31
LL psychological limit	167.5	172.5
Pasty sign PI	132	141.5
Specific weight of G_s	2.11	2.02
Water absorption percentage (%)	360	425
Color	light brown	White

3.2. Pure bitumen

In this research, the pure oil bitumen 60-70 produced in J oil refinery was used to produce clay emulsion, which its specifications are listed in Table (4).

Table 4. The results of the quality control test of the bitumen used in the emulsion production

Experiment test	Standard	Limit	Result
Degree of softness at 25°C, 0.1 mm	ASTM D 5	60-70	68
Soft spot (ring and ball), Celsius	ASTM D30	45-52	51
Ductility at 25°C, 5 cm/min	ASTM D 113	100	100
Specific gravity at 25°C	ASTM D70	1.01-1.06	1.013

3.3. Carboxyl methyl cellulose (CMC)

Carboxyl Methyl Cellulose (CMC) is an anionic polymer dissolved in the water which is made of cellulose which under the normal conditions is white, odorless, colorless, water-suspended and non-fermentable [15]. This material despite of concentration, adhesion and strengthen properties, it also is a factor of dispersion, water maintenance, preserving colloidal state, stabilizer, suspension factor and the layer creation factor. Due to properties of the CMC, this material is widely used in the different industries [16]. Here, the production made by "Saveh chemical and industrial research" company is used which its specifications is shown in Table 5.

3.4. Sodium carbonate

Based on the researches done in the mineral emulsifier field, using some sodium carbonate will be useful for improving the emulsion quality [1]. This material which is a kind of weak emulsifier and PH regulator, is widely used in producing various chemical materials. In the present study, the production of the Semnan Sodium Carbonate Company was used which its specifications, based on the company's tests, are shown in Table 5

Table 5. Specifications of the carboxyl methyl cellulose (CMC) [1] and sodium carbonate specification

Type of chemical material	Sodium carbonate	CMC
Chemical formula	Na_2CO_3	$(C_2.H_4.O_3)$ x
Melting point	851ºC	274ºC
Appearance	White crystalline solid powder - odorless	White powder, odorless
Molar mass, Gr/Mol))	105.98	41000
Gs	2.54	-

3.5. Clay emulsion formulation and preparation

In the present study, producing clay emulsion with different bentonite percentages as an emulsifier factor was done using homogenizer device, shown in Fig. 1, in chemical-physical laboratory of Tehran oil industry research institute. The mechanical homogenizer device is designed to mix and homogenizing the materials which produce the emulsion, suspension or complete solution by dispersing the solid particles inside the liquid. For this purpose, this device has wide application in the pharmaceutical, food, health, chemical and textile industries to produce the solutions, material covers, dye and edible products. The mechanical homogenizer device is composed of a set of rotor, stator and blade where the high speed of grippers leads to crush and compress and break the particles which provides the homogenizing the condense and super viscos materials in a very small time period , Of course, this mixer is used in mixing different materials like Sassobite with bitumen [17-18].

Fig. 3. Homogenizer device and works method

To produce this emulsion, the materials include bitumen 60-70 made by Isfahan J oil refinery, bentonite emulsifier, additive materials such as sulfuric acid 10%, sodium carbonate and industrial CMC, shown in Fig. 4, were used. To produce the clay emulsion, first, an aqueous phase mixture includes water and acid and bentonite having thermal range between 75-80°C was prepared, then hot bitumen with approximately 120°C temperature was added when the mixture was mixed by the homogenizer device. To this end, the mixing plate was located on a heater with constant temperature of 75°C inside the homogenizer device.

Fig. 4. devices and materials used in clay emulsion preparation: (a) Carboxyl Methyl Cellulose, (b) PH gauge, (c) Sodium carbonate, (d) Dilute sulfuric acid, and (E) image of the clay emulsion prepared using 5% of bentonite emulsifier of type Be1 called S1.50

4. Analysis of the results and response surface statistical scheme

The response surface statistical method (RSM) was used to optimize the clay emulsion preparation process. A design used here is a central composite design which obtains the most application among the response surface method techniques. The RSM methodology is a set of applicable mathematical and statistical techniques to create experimental models based on the data related to the test design which was designed by Box *et al.* in 1950s. The target in the response procedure designs, is to optimize the variable output (response) which affected by multiple independent variables (input variables) [19]. In fact, the RSM method was developed for the experimental responses model and then was developed for modeling the numerical tests [20]. In RSM, it is assumed that the errors are random. RSM is widely used for design optimization, reducing the costs of costly analytics methods and their related numerical disorders. In RSM, the convergence is toward the optimal element, because they reduce the disorder factors.

Based on the results obtained from the statistical analysis and the researches done previously, which were described in previous sections, then type of the bitumen emulsions using the percentages of 3, 3.5, 4, 4.5 and 5 of two kinds of bentonite were prepared as mineral emulsifiers. The prepared emulsions were numbered based on their specifications, for example, the S2 code is the prepared specimen from bentonite type Be1 and number 35 indicates the 3.5% of bentonite used as emulsifier. Table 6 shows the material type and the amount of each material which used is preparation of each emulsion specimen.

According to the plots obtained from the statistical analysis, the quality of the emulsion produced is related to the PH of the external phase of the clay emulsion before addition of bitumen, and the viscosity of the produced emulsion is related to the percentages of the bentonite emulsifier. By plotting the weight percentages of the remained emulsion particles on the sieve 850, the amount of the optimal PH was obtained approximately 4.3. Fig. 5. shows the relation between PH, bentonite percentages and viscosity.

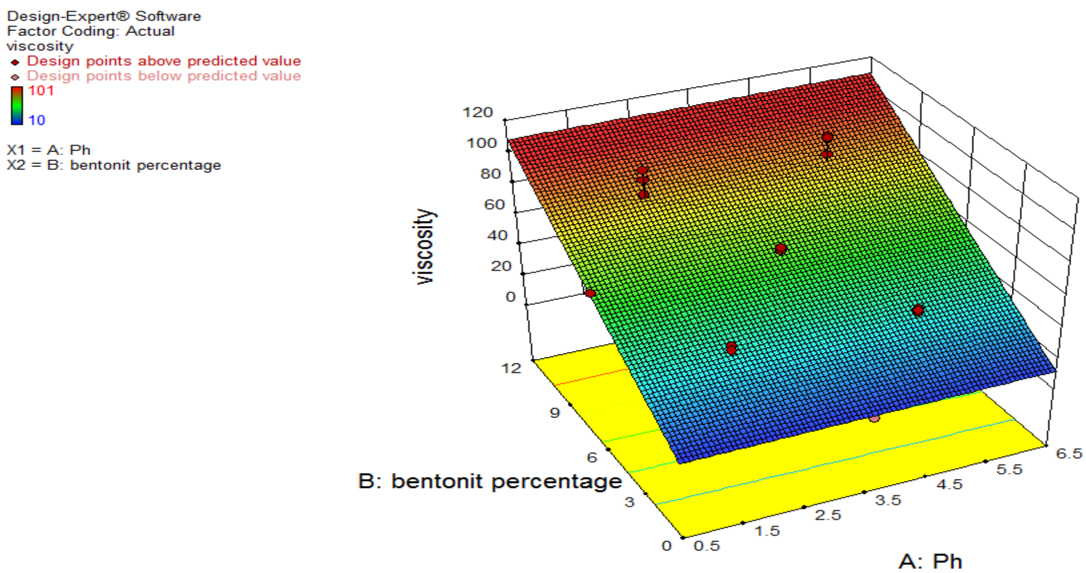

Fig. 5. The plot of the clay emulsion viscosity in terms of the bentonite percentage and pH

According to this plot, an increase in bentonite percentages will cause an increase in viscosity of the saybolt furol emulsion but PH variation of the external phase has not considerable effects on the viscosity. Fig. 6 shows the relation between bentonite and the remained percentage on the sieve 850 μm. According to this figure, by increasing the bentonite percentage, the remained percentages on the sieve increases which is probably due to the agglomeration of bentonite particles. For average values of bentonite (3 to 6%), by increasing the PH up to about 4, the value of the particles remained on the sieve 850 μm reduces, in other word, the quality of the emulsion improves but for higher values, the pH increases. Based on this plot, the optimal pH value for producing the clay emulsion 4.3 is defined so that to be considered for all emulsion which will be produced. It is worth nothing that to equalizing the amount of pure bitumen exist in the observation emulsion and prepared emulsion and to cover the same bitumen in the asphalt specimens which will be prepared later, the percentage of bitumen is considered constant and equal to 50 times the weight percentage of the emulsion.

Table 6. Components of the prepared clay emulsion specimens

Sample number	Percent water	Bentonite Be1%	Bentonite Be2%	Sulfuric acid	Sodium carbonate
S1.30	46.855	3.0	-	0.1	0.045
S1.35	46.30	3.5	-	0.15	0.05
S1.40	45.75	4	-	0.20	0.05
S1.45	45.245	4.5	-	0.20	0.055
S1.50	44.465	5	-	0.3	0.055
S2.30	46.8	-	3.0	0.15	0.050
S2.35	46.30	-	3.5	0.15	0.050
S2.40	45.80	-	4	0.15	0.050
S2.45	45.23	-	4.5	0.22	0.050
S2.50	44.60	-	5	0.30	0.055

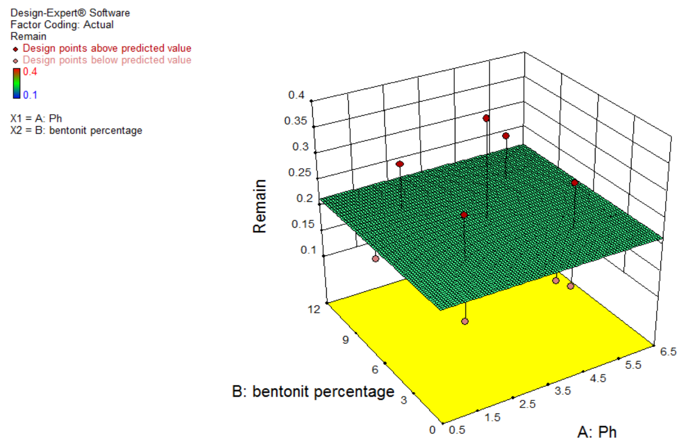

Fig. 6. The plot related to the remained percentages on the sieve 850 μm in terms of bentonite percentage and pH

The emulsion specimens produced before they used in the asphalt cold recycling they were tested using the saybolt furol viscosity, stability and electrical loading test. Based on the results of the electrical loading based standard ASTM D244, the bitumen emulsions stabilized with bentonite have negative surface electrical force with low intensity, because after 30 minutes from beginning of the testing, the current intensity will not be decreased to 2mA and a thin layer of the bitumen sediments on the positive pole. The results related to stabilization and viscosity experiments of the produced emulsions are shown in Fig. 7 and Fig. 8.

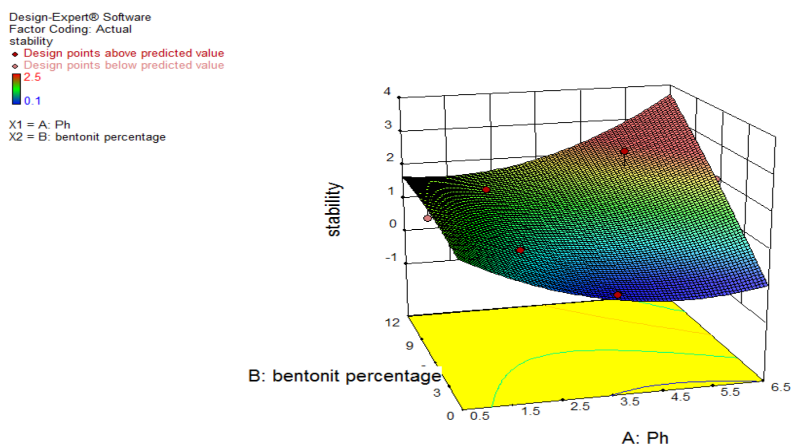

Fig. 7. 24 hours Setting stability test of emulsions for specimens produced of different percentages of bentonites Be1 and Be2

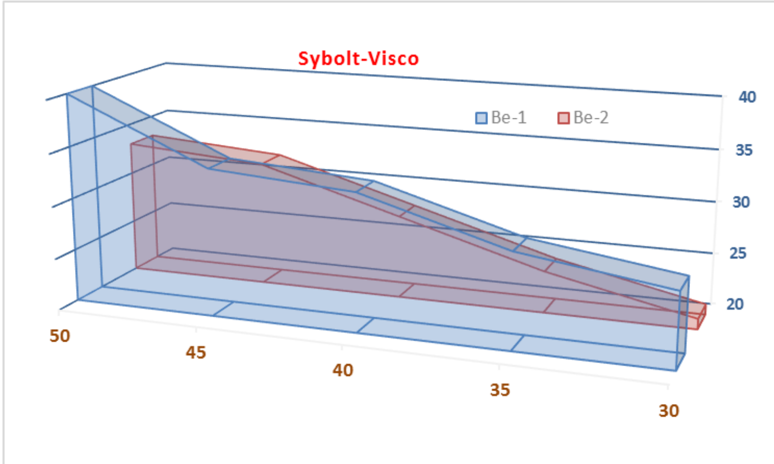

Fig. 8. Results of viscosity test on the produced clay emulsion specimens

Based on the Fig 8 by increasing the bentonite percentages exists in the produced emulsions of both bentonites, the difference of the reduction of upper and lower specimens of the emulsion settling stability test after 24 h, undergo a decreasing trend, in other word, by increasing the amount of bentonite exists in the emulsion, the stability increases. It should be remind that based on the bitumen emulsions instruction of use in the road construction, the allowable value for settling test after 24h is 0.001 percent [21].

5. Conclusions

Today, because of increased costs of the construction and maintenance of the roads, difficulties in preparing good materials as well as shortage in financial resources, recycling the old pavements have been take in to considerations by relevant organizations. Based on the previous researches done, one of the additives which has shown so many positive effects on the emulsion parameters is the clay compounds. The most important results obtained in the present study are summarized as follows:

The bentonite emulsifier can be used to produce the high-stable clay emulsions and to produce cold asphalt mixtures. The clay emulsions have the negative electrical force nature, and this show that the bentonite particles by surrounding the bitumen particles and creating a negative charge repulsion force, makes the emulsion to be more stabilized and creates semi-anionic properties in these types of the emulsions. Given the obtained optimal pH, the clay emulsions have acidic properties.

The viscosity of the produced clay emulsions is in the allowed limits in the road construction. Given the construction materials, clay emulsions have more limited diversity than other bitumen emulsions. The anionic emulsion only react with lime stones and are broken, therefore, the application of theirs emulsion has more limitation that cationic emulsions.

Since the lower the value of the emulsion saturation, the greater its storage capacity and transportability as a result, the clay emulsions have higher storage capacity and longer storage time. The statistical investigations done on the clay emulsions prepared in this paper, the emulsions produced with 3.5 percent of bentonite type 2 has totally more desirable performance than the other clay emulsions.

In order to do more researches on the field of the present research, it is recommended that perform a study to investigate the effect of clay emulsion application on soil stability as well as solutions for improving the performance of clay emulsion under moisture conditions using cement and lime.

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