

Assessment of Removal of Fines from Reclaimed Asphalt Binder Using Fourier Transform Infrared Spectroscopy

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ABSTRACT

The natural environment is suffering greatly from population growth, industrialization, and waste production. When pavements are to be repaired or reconstructed, the waste material poses a threat as it is landfilled and not used up to its potential. Reclaimed asphalt pavement (RAP) has been gaining importance as there is a depletion of natural resources as well as its potential to perform well if reused or recycled. The extraction and recovery process is carried out to evaluate the properties of the aged binder in RAP. Extraction and recovery need to be coupled with gravitational sedimentation and filtration for the removal of mineral fillers from the aged binder + solvent solution. This study uses Centrifuge extraction and a rotary evaporator for extraction and recovery, respectively. In this study, three different solvents, namely tri-chloroethylene (TCE), toluene, and n-propyl bromide (nPB) are used for the centrifuge extraction. Extraction was followed by gravitational sedimentation and filtration. The results were evaluated by Fourier transform infrared spectroscopy (FTIR). Gravitational sedimentation is carried out and samples are tested after 0, 30, 60, 120, 240, and 1440 min by FTIR followed by filtration. In order to assess the properties of aged binder accurately, it should be free from mineral fines. Based on the properties of aged asphalt, percentages of RAP binder and rejuvenator by virgin binder can be evaluated. The results demonstrated that the removal efficiencies were affected by the type of solvent used for extraction.

Keywords: Reclaimed asphalt pavement, FTIR, extraction, recovery, fines, solvent.

INTRODUCTION

Infrastructure growth is a major parameter of showcasing the development of a nation. The road network in turn indicates the progress of a nation. The infrastructure industry is a major contributor to a nation's GDP. The road transportation system in India is extensive, boasting an impressive 144,955 kilometres of National Highways (NH) in 2022–2023. The repair and maintenance of roads lead to waste being produced. Also, the non-renewable materials required for road construction are scarce now, hence there is a need to reuse and recycle the reclaimed materials rather than dumping them away [1]. The reclaimed materials have the potential to be reused and recycled which also makes it cost-effective along with environmental sustainability [2].

The reclaimed binder and aggregates can be used in addition to some additives and rejuvenators to regain their lost strength [3,4]. By employing RAP, not only did the expenses of the entire task decrease, but it also ensured the optimal utilization of available resources. Hence, the goal of creating environment-friendly roads, developing futuristic smart cities, and rehabilitating and upgrading sub-standard roadways can be achieved by embracing various recycled materials; one of them being RAP [5]. In Structural Engineering, after the service life ends, structures are retrofitted using Fiber reinforced plastics to regain their usability [6]. Similarly, for road construction reclaiming is a feasible solution to it. A lot of study has already been carried out on virgin asphalt using different tests such as the Indirect Tensile fatigue test to determine the fatigue life of a pavement [7]. Hydrated lime is also used in pavements for increasing moisture

susceptibility [8]. A lot of research is being carried out on using RAP and its rheological properties to study the extent to which it can be used [9, 10].

To reuse or recycle the reclaimed material, the extraction and recovery of asphalt is necessary. This necessity arises to access the properties of the reclaimed asphalt and provide suitable treatments to it, so that it can perform better in its lifecycle [11, 12]. As RAP is being widely used in practice nowadays, the need for extraction and recovery of asphalt binder has escalated [13]. RAP undergoes hardening which is termed ageing during its lifecycle. It is exposed to environmental and traffic conditions, which causes physical and chemical changes. The changes are imparted due to heat, oxygen, sunlight, and water [14]. Also, to check and assure the quality of binder used on-site, extraction and recovery plays a major role. Due to oxidative ageing, the rheological properties of bitumen are affected [15].

For extraction, centrifuge, vacuum, and reflux methods can be used. In the reflux method of extraction, the binder is heated which causes it to age, but the binder content can be accurately calculated by this method. Being performed at high temperatures adds risk to the operator. The vacuum extraction method is good for determining the binder content but is very difficult to clean and hence is not widely used. The centrifuge extraction is performed at room temperature and does not heat the binder and hence is widely used. As the reclaimed asphalt is to be obtained for our inspection, the best-suited method is centrifuge extraction. Similarly for recovery, Abson and rotary evaporator can be used. The Abson method leads to ageing of binder and residual solvent is present in the binder. To overcome this, the rotary evaporator method is used [16]. The selection of solvents for extraction also plays an important role [17].

After centrifuge extraction, mineral fines are present in the solvent + binder solution. The presence of mineral fines can be seen in the FTIR spectrum [18]. FTIR is also an efficient technique to access the ageing of binders [19–21]. The short term ageing and long term ageing of bitumen and modified bitumen can be identified based on the sulfoxide and carbonyl indices which can be identified and calculated using FTIR [22]. For removal of fines, gravitational sedimentation, filtration, and centrifugal sedimentation can be used. Filtration alone, takes a long period. In centrifugal sedimentation, only a small amount of sample can be used and is not feasible on the field. Gravitational

sedimentation can be incorporated to overcome these drawbacks [23]. The mineral fines comprise inorganic oxides, with silicon dioxide (SiO_2) as its major component (around 60%) with a wave-number near 1000 cm^{-1} [24]. The minor components of mineral fines include Magnesium oxide, Aluminium oxide, Calcium oxide, etc.

In this research, three different solvents are used for the centrifuge extraction. The efficiency of the solvents is determined by the binder content obtained. For the removal of mineral fines, gravitational sedimentation is employed, and samples are collected at different heights and time intervals. The FTIR analysis of the samples is performed to access the presence or absence of mineral fines in the sample. In FTIR analysis the absorbance is calculated from the percentage of transmittance. It is followed by filtration and finally reclaimed asphalt binder is recovered by rotary evaporator. In this study, RAP material is collected from the field and experimentation is done, unlike most literature in which RAP is prepared by simulation conditions in the laboratory.

MATERIALS AND METHODOLOGY

Reclaimed asphalt pavement

RAP was collected from the top 50 mm layer of a bituminous pavement near Dehere, Ahmednagar-Manmad Highway, Maharashtra, India which was constructed 7 years ago. It was originally constructed with VG30. This road is under repairs and maintenance because of damages due to heavy traffic conditions.

Solvents

Tri-Chloroethylene, Toluene, and n-Propyl Bromide were used for the Centrifuge Extraction Process to dissolve the asphalt binder.

Centrifuge extraction

The obtained RAP material was heated in an oven at $110\text{ }^\circ\text{C}$ to remove moisture and also make it easier to crush the material. This crushed RAP was measured to 1 kg and was transferred to the centrifuge vessel. For 1 kg of RAP, 600 ml of solvent was poured, and it was allowed to soak for 1 hour. The vessel is rotated at a maximum speed of 3600 rpm to obtain the aggregates and solvent binder solution separately. The procedure is followed as per [25].

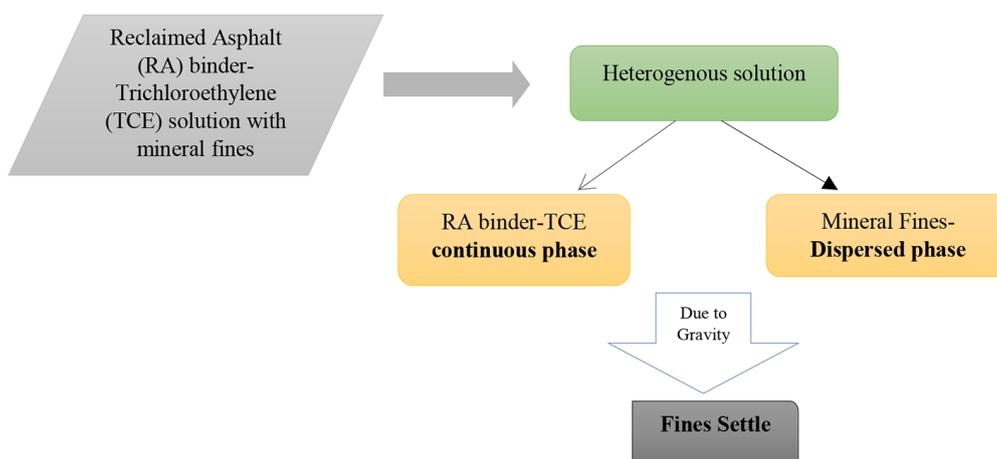


Fig. 1. Schematic of gravitational sedimentation

Gravitational sedimentation

In order to remove the fines from the reclaimed binder+ solvent solution, gravitational Sedimentation was employed followed by filtration as it can be used on a large scale. A schematic of gravitational sedimentation is shown in Figure 1.

A 10 mm radius tube was used, and the height of the sample kept for gravitational sedimentation was 100 mm. For uniform dispersion of mineral fines, the tubes were rotated for a period of 1 minute. The height of the sample is h , and it is divided into 2 parts of height $h/2$. The Samples were collected from three heights from the top of the sample, 0, $h/2$, and h as shown in Figure 2. Nomenclature given to the samples from the top of the sample is S1 for 0, S2 for $h/2$, and S3 for height h . Samples were collected in vials with pipette of 1 ml. A similar procedure was repeated for different intervals of time. The time intervals selected were 0, 30, 60, 120, 240, and 1440 min. This procedure was repeated for all three solvents. 54 samples were collected, 18 for each solvent. While drawing the samples via pipette, it was made sure that the tubes were undisturbed.

Filtration

The sample obtained at height $h/2$ from the top of the sample level was filtered from a filter paper with an aperture of 70–90 microns. The filtrate was collected in a glass tube. This filtrate is free from any mineral fines.

Recovery by rotary evaporator

The filtrate obtained after filtration was subjected to a rotary evaporator [26]. Only the

sample of height $h/2$ from the top was used for the rotary evaporator based on the results of FTIR for gravitational sedimentation. The pictorial representation of the methodology followed is given in Figure 3.

Fourier transform infrared spectroscopy

FTIR test was performed at the Central Instrumentation Facility of the Chemistry Department of the Savitribai Phule Pune University, Pune, Maharashtra, India. The spectra of samples collected after gravitational sedimentation was obtained by subjecting the samples to FTIR spectroscopy. The spectrum of mineral fines was also found. The Si-O bond which is dominant in mineral fines is used to study the presence or absence of fines in the samples. The intensity of the SiO_2 peak (around 1000 cm^{-1}) is determined and based on the results, the presence and absence of fines are stated.

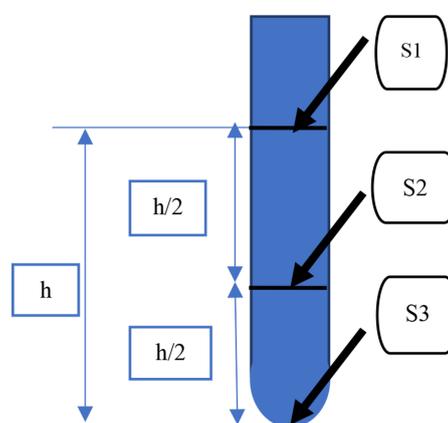


Fig. 2. Sample collection of gravitational sedimentation

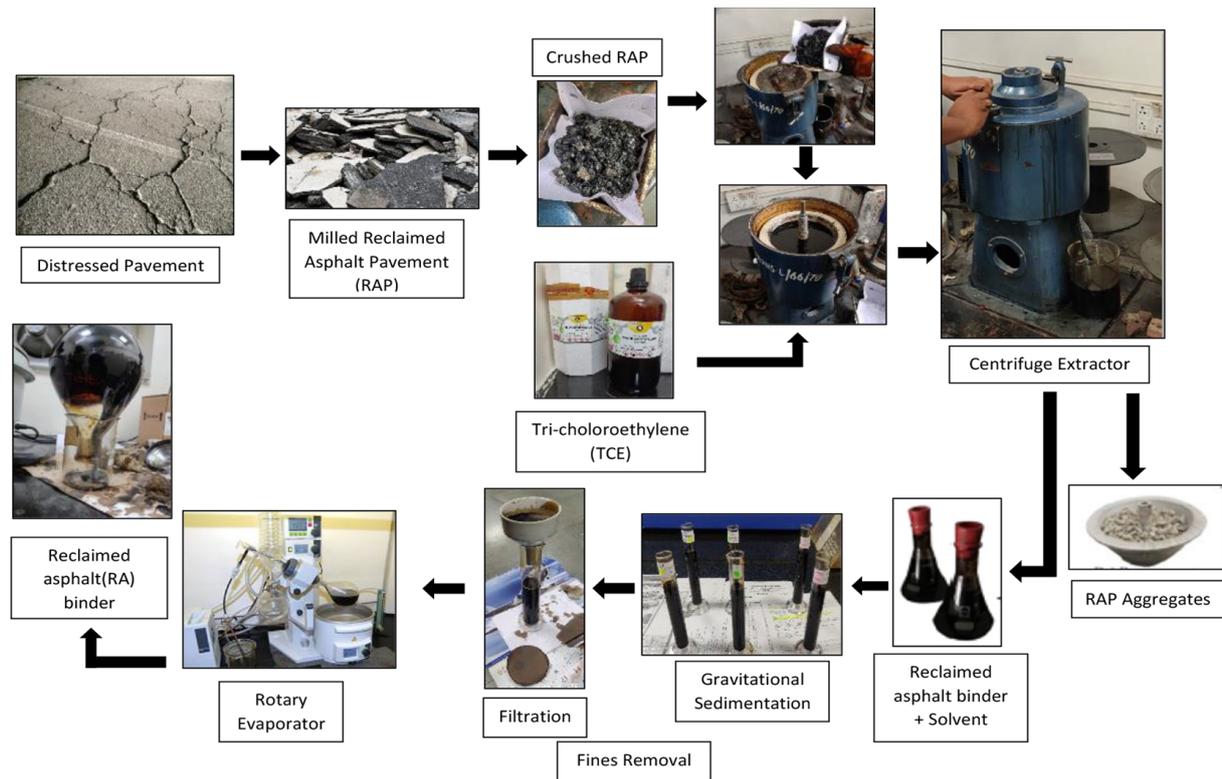


Fig. 3. Methodology adopted

RESULTS AND DISCUSSION

Binder content

The binder content was determined for the reclaimed asphalt pavement sample after centrifuge extraction as per ASTM D2172. Three trials for each binder were conducted. The results are shown in Figure 4. The average binder content (%) for the same RAP material is 4.95% for TCE, 4.70% for nPB, and 4.19% for Toluene. The results indicate that dissolution was thorough in the case of TCE. Results for nPB are in the range of TCE but for Toluene the results have a higher variation. TCE

gives the best results in terms of binder dissolution without leaving traces of binder on the aggregates and hence explains its acceptance in the industry.

FTIR samples

The FTIR spectroscopy of fines was carried out and the dominant peak was found at a wave-number of 994 cm^{-1} . This peak resembles the presence of SiO_2 in the fines. The absorbance value of this peak was observed to be 2.186. In this section, the spectra of S1 samples after different time intervals are plotted on a graph to analyze the

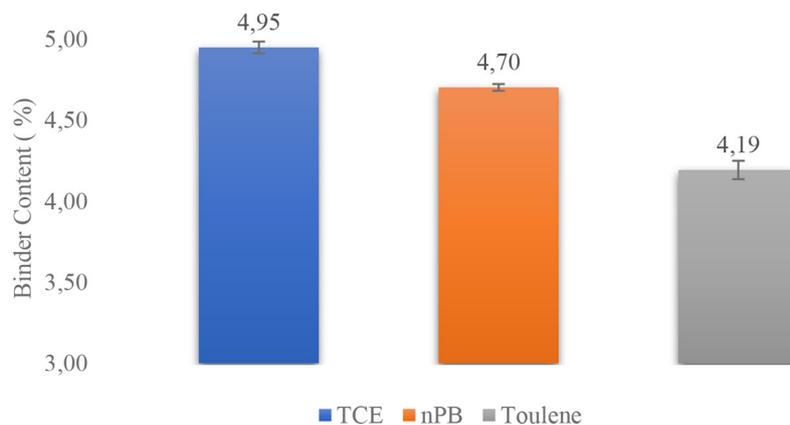


Fig. 4. Binder content for different solvents

results as shown in Figures 5, 8, 11. Similarly, the S2 as shown in Figures 6, 9, 12 and S3 as shown in Figures 7, 10, 13 samples are plotted together. There is a slight shift in the peak of the Si-O bond for TCE solutions in comparison to that of fines. This happens due to the physical and chemical interaction. In the solution, the peak is found at 1014 cm^{-1} . For the S1 sample of TCE, the Si-O peak observed at 0 min was recorded to have an absorbance of 2.135. After 30 mins of sedimentation, the peak dropped considerably to an absorbance of 2.0224. There was a further reduction in peak after 1 h of gravitational sedimentation to a value of 2.0136. For the time intervals of 2 h, 4 h, and 24 h, the peak reduced slightly to 2.0094, 2.0084, and 2.0084. The values of 4 h and 24 h are coinciding and thus it can be concluded that for TCE, gravitational sedimentation yields same

results after 4 h which means the sedimentation of fines is almost complete after 4 h. The peaks were reduced by 5.93% from 0 mins to 24 h. This is shown in Figure 5.

For S2 samples of TCE, i.e. at h/2 height, the peaks at 1014 cm^{-1} are 2.138, 2.019, 2.012, 2.0094, 2.0084, 2.0069 respectively for 0 min, 30 min, 1 h, 2 h, 4 h and 24 h. The dropping of the peak was significant till 2 h interval, but after that, the peaks are very close as given in Figure 6.

For the S3 sample, the peaks will increase as the time advances, as the mineral fines will settle down in this region. From the FTIR spectrum of the S3 sample in Figure 7 of TCE, it can be seen that the peak for 0 min time interval is 2.0057, for 30 min is 2.0076, for 1 h 2.0361, for 2 h 2.0388, for 4 h 2.0568 and 24 h the peak rises to 2.1379. This rise is very high at 24 h, hence the bottom

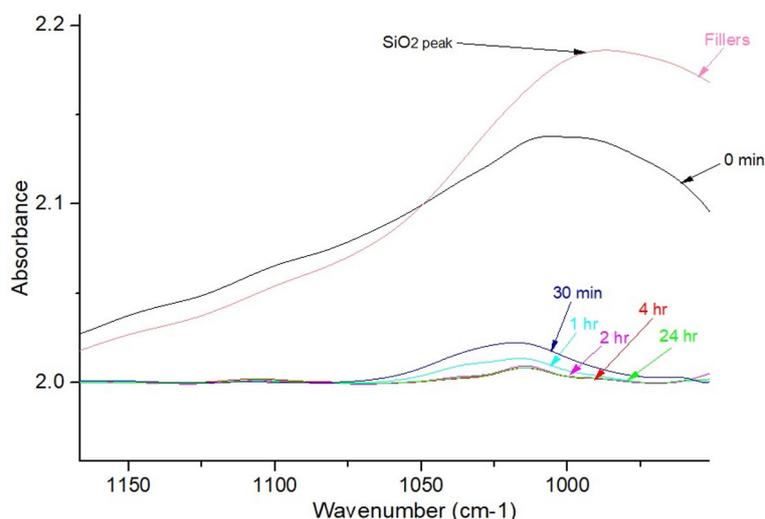


Fig. 5. FTIR Spectra of TCE, S1 sample

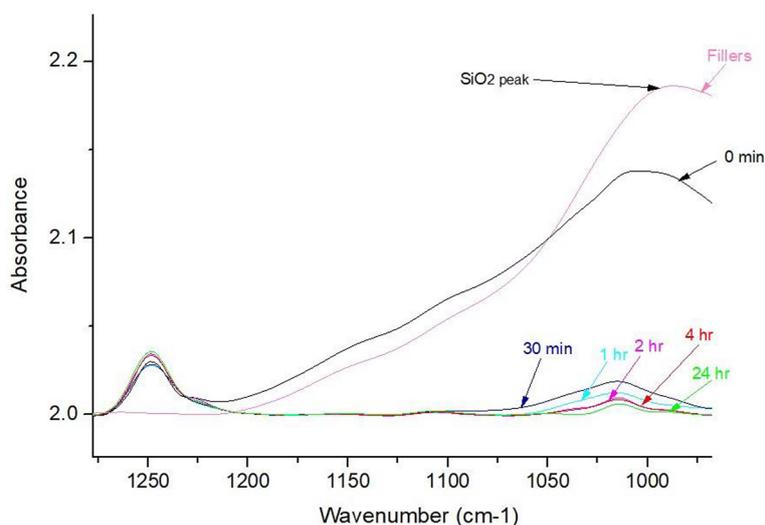


Fig. 6. FTIR Spectra of TCE, S2 sample

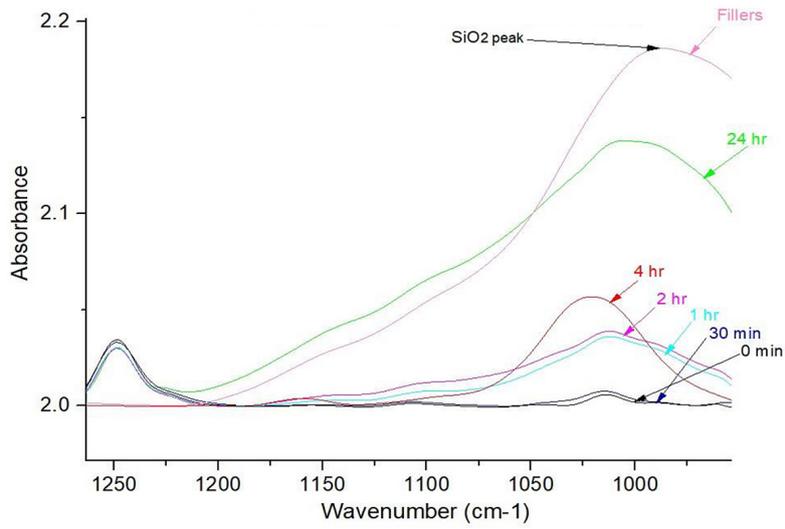


Fig. 7. FTIR Spectra of TCE, S3 sample

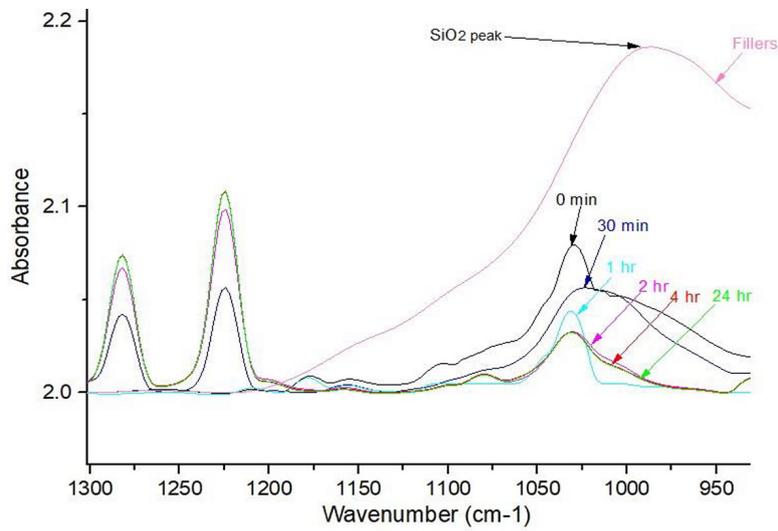


Fig. 8. FTIR Spectra of nPB, S1 sample

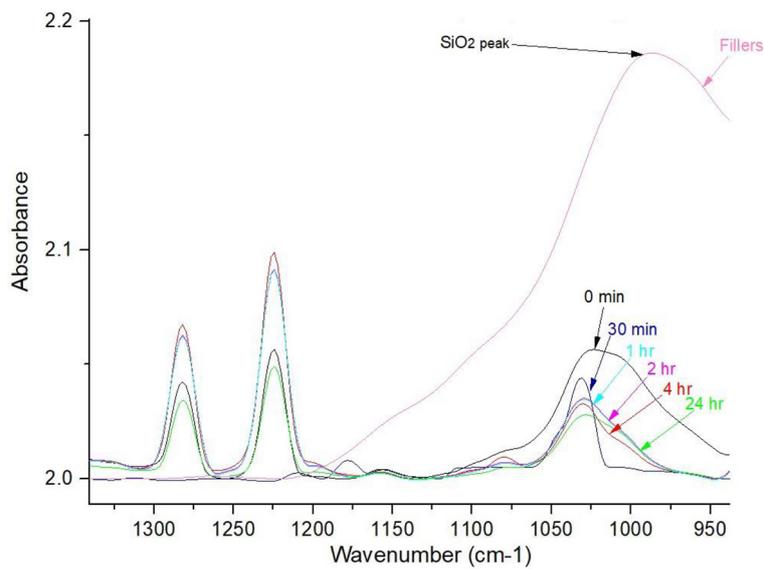


Fig. 9. FTIR Spectra of nPB, S2 sample

half sample shows large sedimentation as time progresses and hence this sample should be discarded. The top sample of height $h/2$ should be used for recovery.

For S1 samples of nPB, the peaks are 2.0795, 2.0564, 2.044, 2.0328, 2.0328, and 2.0324 for 0 min, 30 min, 1 h, 2 h, 4 h and 24 h respectively. This implies that the decrease in intensity of peaks is lesser when compared with TCE sample S1. After 2 h sedimentation, the reduction in peak intensity is very low as seen in Figure 8. The peaks almost coincide for 2 h, 4 h and 24 h time interval and no significant reduction can be seen. The peak intensity dropped down by 2.26% from 0 min to 24 h.

For S2 samples of nPB, the peaks are 2.0564, 2.044, 2.035, 2.035, 2.0328 and 2.028 for 0

min, 30 min, 1 h, 2 h, 4 h and 24 h respectively. There has been a slight decrease in the intensities of the peaks up to 24 hrs as given in Figure 9. For S3 samples of nPB, the peaks are 2.0245, 2.0313, 2.0315, 2.0319, 2.0564 and 2.0741 for 0 min, 30 min, 1, 2, 4 and 24 h respectively as shown in Figure 10. This implies that the sedimentation takes place up to 24 h and hence the sample at the bottom should not be used for recovery of asphalt binder as it contains mineral fines.

For S1 samples of Toluene, the peaks are 2.1086, 2.0399, 2.0374, 2.0371, 2.03254 and 2.0315 for 0 min, 30 min, 1 h, 2 h, 4 h and 24 h respectively as shown in Figure 11. This implies that the decrease in intensity of peaks

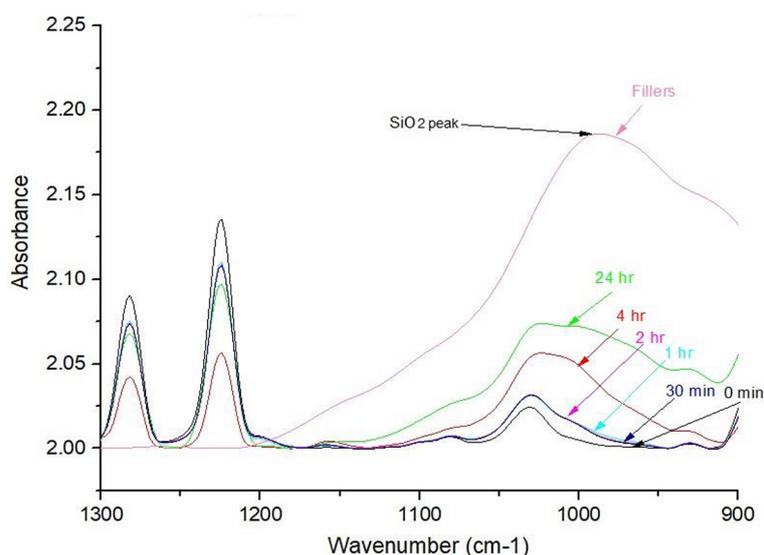


Fig. 10. FTIR Spectra of nPB, S3 sample

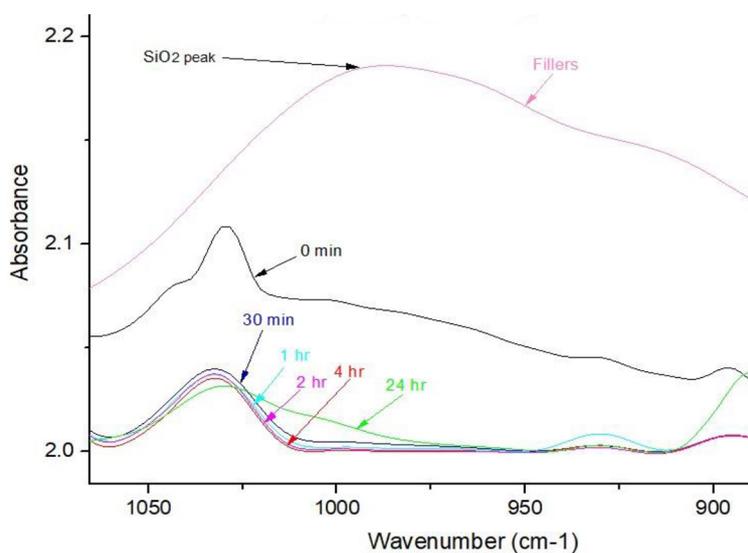


Fig. 11. FTIR Spectra of Toluene, S1 sample

is lesser when compared with TCE sample S1. There is significant difference in the peaks up to 24 h. The peaks reduced by 3.66% from 0 min to 24 h.

For S2 samples of Toluene, the peaks are 2.1086, 2.0399, 2.0374, 2.0371, 2.0354 and 2.0315 for 0 min, 30 min, 1 h, 2 h, 4 h and 24 h respectively. There has been a slight decrease in the intensities of the peaks up to 24 h as seen in Figure 12.

For S3 samples of Toluene, the peaks are 2.0324, 2.0378, 2.0407, 2.0469, 2.0482 and 2.1086 for 0 min, 30 min, 1 h, 2 h, 4 h and 24 h respectively as shown in Figure 13. This implies

that the sedimentation takes place up to 24 h and hence the sample at the bottom should not be used for recovery of asphalt binder as it contains mineral fines. Maximum increase in the peak is observed between 4 h to 24 h.

For samples S1, the peaks of the three solvents namely TCE, nPB and Toluene are 2.0084, 2.0324 and 2.0315 respectively after 24 h of gravitational sedimentation. This indicates that the amount of fines are the least in TCE sample after the sedimentation process. After sedimentation, hence filtration is followed. The maximum removal of fines was found in TCE solvent based upon the peak of Si-O.

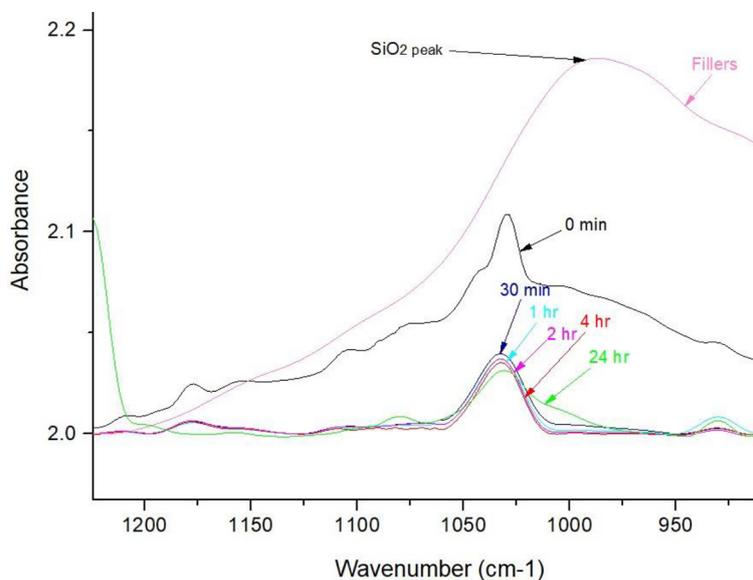


Fig. 12. FTIR Spectra of Toluene, S2 sample

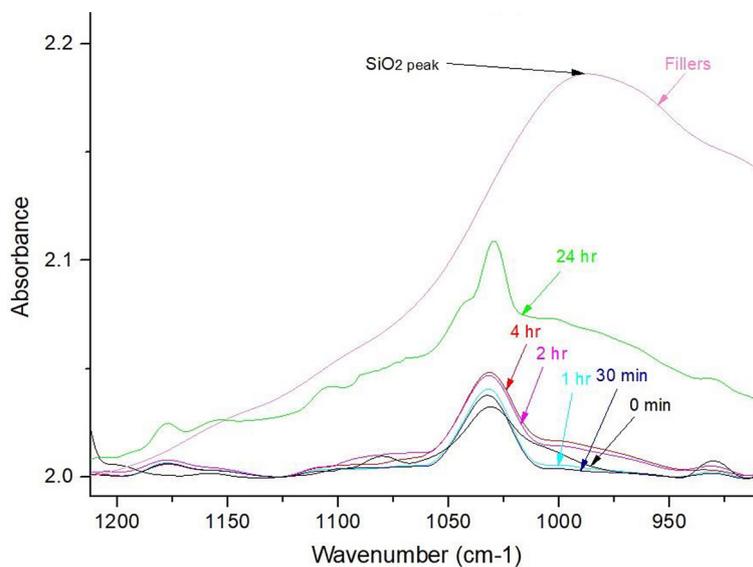


Fig. 13. FTIR Spectra of Toluene, S3 sample

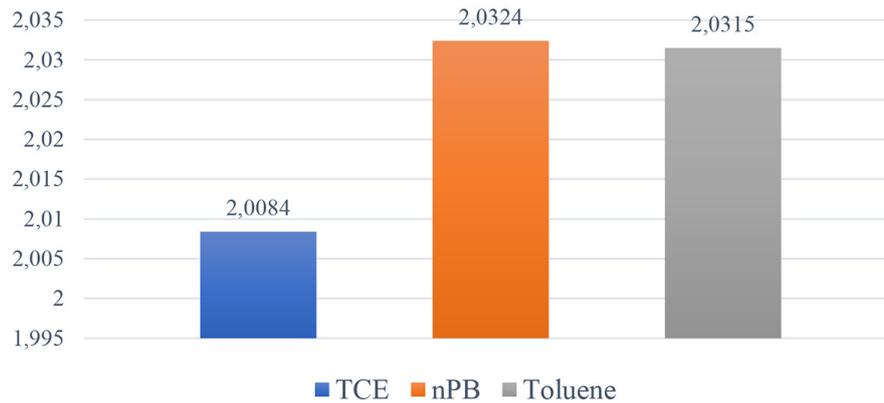


Fig. 14. Peaks of Si-O for S1 samples after 24 hours

CONCLUSIONS

The extraction and recovery of binder play a crucial role to examine the properties of the reclaimed asphalt. The degree of purity and level of accuracy of the reclaimed asphalt depends on the method used for its extraction and recovery. This paper summarizes the method for effective extraction and recovery of binder, free from mineral fines. The conclusions drawn from this study are as follows. The Binder content for the same RAP material was highest when tri-chloroethylene was used as a solvent which implies higher dissolution of asphalt binder in TCE followed by nPB and then Toluene. The FTIR spectroscopy analysis indicates that the Si-O peak of S1 and S2 samples go on decreasing as time progresses and vice versa for S3 samples. Also after 4 h, the peaks in TCE coincide indicating that gravitational sedimentation of 4 h is optimum for extraction by TCE solvent. In case of nPB, significant drop in peaks was observed up to 24 h and the drop was higher compared to nPB, in case of Toluene. Hence, when nPB and Toluene are used as solvents, 24 h gravitational sedimentation should be followed. The peak of mineral fines was observed to be the least for TCE after gravitational sedimentation of 24 h, indicating maximum removal of fines as compared to nPB and Toluene. In terms of removal, TCE gives the best results followed by Toluene and then nPB.

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