

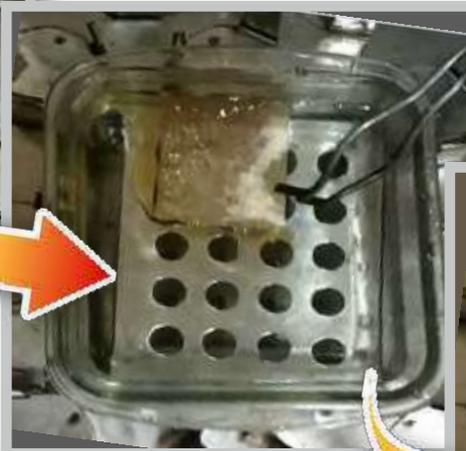
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Water with spilled oil



Soaking of Oil



Soaking Completed



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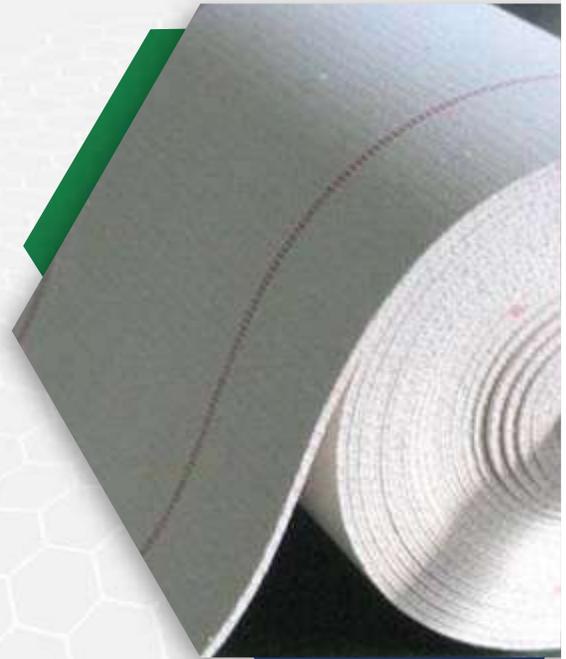
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EDITOR'S DESK

Dear Readers,

Greetings!!

Research with persistent and focused efforts lead to a positive result. Fostering research and providing a platform to publish quality research papers and related articles has been a continuous effort of BTRA Scan. It is time for us to continue work hard to help the journal in climbing up the ranking ladder. In continuation to this effort, I am delighted to present to our readers the 4th issue of 51 Edition of BTRA SCAN.

This issue has papers from the different domains such as evaluation of ground improvement using geosynthetics, cotton waste base super oleophilic and hydrophobic sorbent, Cure kinetics of unsaturated polyester resin. Now we are open for authors from outside so researchers can send their original articles, case studies, research reviews or empirical contributions for publication in our journal.

I thank my associate editor Dr. Prasanta K. Panda and the entire publishing team for all their support. Together we would work towards making the journal a truly influential publication. Comments and suggestions are always welcome.

Our sincere thanks to all the reader and contributors for their support and interest.

T V Sreekumar, PhD

Director, BTRA

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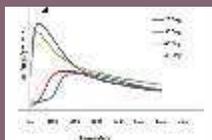
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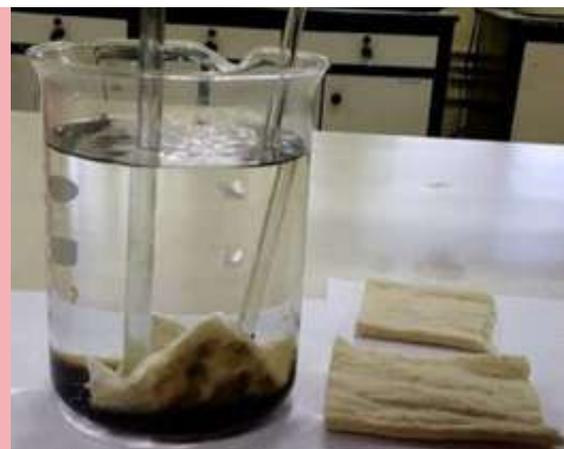
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Development of Bio-degradable Cotton Waste Based Super Oleophilic and Super Hydrophobic Sorbent for Oil Spill Clean-up



M.P Sathianarayanan* & Karishma Hemani

The Bombay Textile Research Association, L B S Marg, Ghatkopar (W), Mumbai 400086

Abstract

Cotton waste, which is generated from various processes in a textile mill like carding, blow room, etc. was collected, cleaned, pre-treated, and processed in a carding machine to make the web. The cotton web was converted to a non-woven batt through a needle punching process. The non-woven fabric was chemically modified by treating it with silica nanoparticles synthesized via the Stober process followed by hexadecyltrimethoxysilane (HDTMS) treatment. The chemically functionalized fabric was showing super hydrophobic and super oleophilic property. The oil absorption capacity of the developed sorbent was 25-30 g/g and the water contact angle was more than 150° . The morphology of the sorbent was characterized by SEM. The sorbent was found to be reusable at least 5-6 times with an oil recovery of more than 60%. Biodegradability of the chemically modified cotton was also studied and was found to be fully biodegradable. The developed sorbent was found to be useful for cleaning oil spillage on the land surface, oil on surface water, and underwater also and was found to be superior to the commercially available synthetic polypropylene/polyurethane sorbent.

Keywords

Cotton waste, Hydrophobic, Oleophilic, Oil sorbent, Sol-gel, Silica nano particle.

Citation

M.P Sathianarayanan & Karishma Hemani - "Development of bio-degradable cotton waste based super oleophilic and super hydrophobic sorbent for oil spill clean-up", *BTRA Scan* - Vol. LI No. 4 October 2022, Page no. 1 to 8

1.0 Introduction

Oil is one of the most important sources of energy and is also used as raw material for synthetic polymers and chemicals worldwide [1-3]. Oil has been part of the natural environment for millions of years. Oil spills into land, river, or ocean during production, transportation, tanker disaster, accidents and imposes a severe environmental problem. Oil spills launch unsafe chemical substances along with polycyclic aromatic hydrocarbons which are toxic to each aquatic life and humans and might also additionally require many years to remove [4-7]. Complete removal of crude oil and petroleum product that are spilled at the sea or water bodies is a more difficult task than cleaning on land surface as part of the oil stays in a colloidal form and remains on the water surface. The basic methods for oil spill collection and clean-up are chemical treatment, mechanical treatment, and biological treatment. Chemical treatments are not so popular

and effective as it leads to secondary contamination. The mechanical methods involve the transfer of oil from spills to temporary storage using oil sorbents. There are various types of oil sorbents being used for oil spill clean-up. Good oil sorbents are characterized by high sorption capacity, good buoyancy, low density, adequate oil retention and re-usability [8]. The most widely used oil sorbent across the world is polypropylene or polyethylene based mat/boom/pillow as it merits all the prerequisites of a good sorbent except the bio-degradability.

Cotton, a natural and bio-degradable cellulosic material, will be an ideal choice for good oil sorbent but its high density and natural hydrophilic property discourages its usage as an oil sorbent, especially on water bodies. Cotton will be extra benefitted when made hydrophobic through chemical modification. Change of cotton to make it super hydrophobic extends the usage of cotton to various end uses like water repellent self-cleansing of fabric and oil spill clean-up in

*Corresponding author,
E-mail: ecolab@btraindia.com

water bodies in which it might repulse water and absorb oil. Good quality cotton has an amazing industrial value in textiles; hence, the use of this value added cotton for oil spill clean-up is not a good option and is commercially no longer viable. Hence, the usage of cotton waste generated from cotton generators of their diverse method like blow room, carding, etc. can be a super choice. In India, high cotton production is accompanied by the generation of tons of waste each year, either from textile mills or cotton fields. These wastes are disposed of through burning, which in flip will increase the carbon dioxide level in the ecosystem which provides worldwide warming and once again it creates pollution. Hence, cotton waste based oil absorbent can have value addition, be economically viable, sustainable product, and biodegradable. Again, the usage of cotton waste as oil sorbent will be a green initiative to control pollution dually. Direct use of cotton fiber as an oil absorbent has many negative aspects as constrained kind of oil absorption, low oil absorption and retention. In addition to this, the inherent water absorption tendency of cotton, which is an undesirable property for oil sorbent, will lessen the oil pickup. To conquer those problems, cotton fibres need to be chemically modified to make them a super- hydrophobic and super oleophilic sorbent. For practical application, loose fibrous sorbents need to be compacted as non-woven mats, or cushions, pads, booms and need to be efficaciously recoverable without prompting secondary contamination so that these sorbents do not disintegrate when applied to oil spill cleanup.

2. Experimental

2.1 Materials, equipment, and methods

2.1.1 Materials

Waste cotton fibers were procured from Tata mills and Techno craft industries, in Mumbai from their various cotton processing sections like carding, combing, blow room, etc. Premium diesel engine oil (API CI-4 15W-40), Diesel oil, and Crude oil (tar) were purchased from a petrol pump, in Mumbai. Ethanol (99.9%, AR grade), Tetraethyl orthosilicate (TEOS, 98%), Ammonia solution (NH_4OH 25%), Methyl triethoxysilane (MTES, 98%), Hexadecyltrimethoxysilane (HDTMS, 85%), were purchased from Sigma Aldrich.

2.1.2 Equipment

Analytical balance (Shimadzu AUX 220), Carding Machine (Dormetx Cormatex Italy), Needle punching machine (DILO – Germany), Scanning electron microscope SEM (JSM IT200 Joel), Easy Drop Analyzer (Stringray & Kruss), Microscope, Mechanical Shaker, were utilized from within BTRA facility.

2.1.3 Preparation of non-woven fabric from cotton waste

Raw cotton waste procured from different textile mills was cleaned in a trash separating machine (Stratex Trash

separator) where all dust dirt and non-fibrous contamination are removed. The cleaned fibre was fed into a mini carding machine which individualizes the fibers and aligns them in one direction to develop a cotton web. The purpose of carding was to disentangle and mix fibers to form a homogeneous web of uniform weight per unit area. This was carried out in a series of fiber opening and layering actions accomplished by the interaction of tooth rollers situated throughout the carding machine. The cylinder is the heart of the carding machine and is the central distributor of the fibers during the process. The doffer rollers condense and remove fibers from the cylinder in the form of a continuous web. Carding of cotton fibres was done in Cormatex Prato mini carding machine, wherein the fibre tufts were converted into a carding web. The carding web was passed through a cross lapper to a needle punching machine to form a batt of 200 GSM. Needle punching process is based on subjecting a web in the needle punching machine to the effect of the needles oscillating vertically, slanting, or both directions of the surface of the web. As the needle penetrates the web, there barbs or grooves catch the fibers and draw them in a vertical or slanting direction through the thickness of the web.

We have prepared non-woven fabric by using vertical needle punching, where it was done from one or both sides. Needle punching on both sides was arranged in such ways that the needle heads are located on both sides of the needle web. The modern variations of this type of needle board arrangement in most cases make possible needle punching both with in-stroke and off-stroke movement of needle boards. During the counter movement of the needle boards, the needles enter the material from both sides at the same time. To make this possible, each needle board carries a number of needles equal to half of the matching holes in the stripper and bed plates. The holes for both needle boards are spaced uniformly, in most cases, following the pattern of the plain weave. In this case, the needles on the upper board enter the web as the needles on the lower board leave it, and vice versa. The parameter of needle punching machine was fixed to get 300-600 strokes per minute with a draw of 1.1 m/min.

Fig.1 and Fig.2 are photographs of cotton waste and cotton waste based non-woven fabric respectively.



Fig-1 photograph of cotton waste



Fig-2 photograph of nonwoven fabric

2.1.4 Chemical modification of non-woven cotton fabric

Silica nanoparticles were synthesised in the laboratory via the Stober process (sol-gel). The synthesized nanoparticles were sprayed on both sides of the non-woven fabric by using a spray gun and the fabric was dried at 80° C for 10 minutes. The dried fabric was further treated with HDTMS (3 % in ethanol) by using a spray gun. The treated fabric was dried at 80° C and cured at 150° C for 10 minutes. The entire chemical reaction on cotton takes place in two stages.

When a sol-gel is applied to cotton fabric, the nano - SiO₂ particles are deposited on the surface of the cotton fabric. The condensation reaction between the -OH group from the surface of cotton fabric and the Si-OH group from the nano-SiO₂ particles bonding takes place during the drying process. As the nano- SiO₂ covalently bonded onto the cotton fabric, silica particles deposited on the surface increases the hydrophobic effect of the textile material by decreasing the surface energy. The -CH₃ bunch blesses the SiO₂ particles with hydrophobicity and oleophilicity. The combination of nano- roughness of nano- SiO₂ and the inherent micro-roughness of fabrics created a nano-binary surface roughness on cotton fabric's surface, which would greatly enhance the wetting behaviour of the surface of fabrics. In the second stage, sol-gel treated fabric was further treated with HDTMS, wherein HDTMS is hydrolysed in an aqueous ethanol solution to form alkylsilanol. The self-assembly of HDTMS is formed by the reaction between the alkylsilanol and the surface hydroxyl groups of silica nanoparticles on the cotton fabric surface.

2.1.5 Measurement of oil and water absorption properties of sorbents

There are two standard test methods (ASTM F 726) for the evaluation of the oil and water uptake of sorbents. They are static and dynamic tests. In the static condition, the sorbent (4.0 g) was kept in a tray filled with oil, after 15 minutes, the sorbent was removed from the oil and drained for 30 seconds, and weighed. The oil absorption capacity of the sorbent (g/g) was calculated from the initial weights and the final weight of the sorbents.

In dynamic conditions, Synthetic seawater was prepared by dissolving 35g of NaCl in 1000 ml distilled water. A known weight of sorbent was placed in a tray that contains artificial seawater (500ml) and 50 g of crude oil (Mark diamond 15w-40 Premium diesel engine oil APL Cf4). The density of oil used was 0.87 grams per cubic centimeter. 4.0 g of sorbent was placed in the tray and shaken in a laboratory shaker at a frequency of 150 cycles/minute for 15 minutes. The sorbent was held for 30 seconds to drain out excess oil /water and weighed. The oil absorption capacity of the sorbent (g/g) was calculated from the initial weight and final weight of the sorbent. The water absorption property of the sorbent was also measured in a similar manner but using only water and without oil.

$$\text{Absorption capacity (g/g)} = \frac{(M_f - M_o)}{M_o}$$

Where M_f is the weight of oil soaked material and M_o is the initial weight of the material.

3. Results and Discussion

3.1 Absorption properties of the sorbent

The oil and water absorption property of the chemically modified cotton sorbent of various cotton wastes was studied with motor oil and diesel engine oil. Table-1&2 shows a comparative study of the oil and water absorption capacity of sorbents prepared from carding waste and blow room waste against commercially available polypropylene and polyurethane sorbent. It was observed that under static conditions carding waste absorbs 30 to 30.3 g/g of motor oil and under dynamic conditions, it was 28.2 to 29.1g/g. Similarly blow room waste sorbent, under static conditions motor oil absorption capacity was 21.3 to 25.9 g/g, and that of dynamic conditions the same was 20.2 to 27.2 g/g. At the same time, commercial polypropylene sorbent, motor oil absorption capacity for static and dynamic conditions was 12.3 g/g and 12.7 g/g respectively. For polyurethane sorbent, motor oil absorption capacity for static and dynamic conditions was 10.9 g/g and 14.2 g/g respectively. This indicates that modified cotton waste-based sorbent has a higher oil absorption capacity than polypropylene and polyurethane sorbent. The water absorption property of polypropylene and polyurethane was 0.1g/g and 1.7 g/g respectively and that of cotton waste sorbent was 0.01 to 0.18 g/g which shows chemically modified cotton sorbent is more hydrophobic than polypropylene and polyurethane. A similar trend was also observed in the case of diesel oil in water (Table 2). This indicates cotton, which is hydrophilic has become hydrophobic after chemical modification and is in line with hydrophobic polypropylene. Since the viscosity of diesel oil is lower than motor oil, oil pick up was also lower. Modified cotton sorbent absorbs 2 to 2.5 times more oil than polypropylene fabric. At the same time, the water absorption capacity of both polypropylene and cotton sorbent was almost the same.

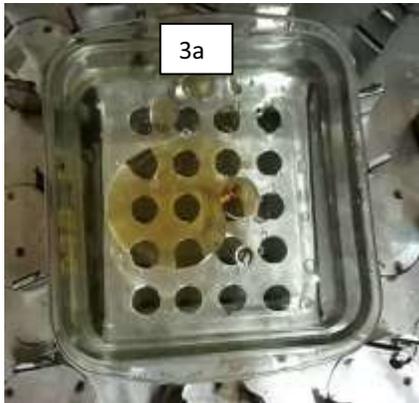


Fig.3a Oil floating on water surface

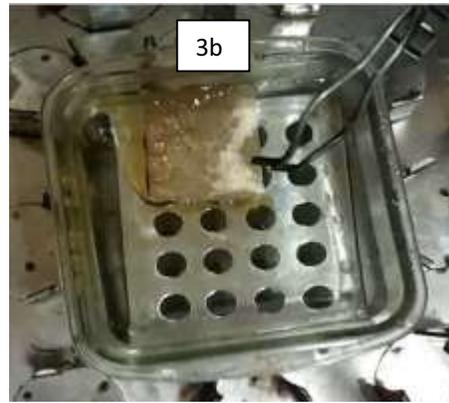


Fig.3b Sorbent absorbs oil from the water surface

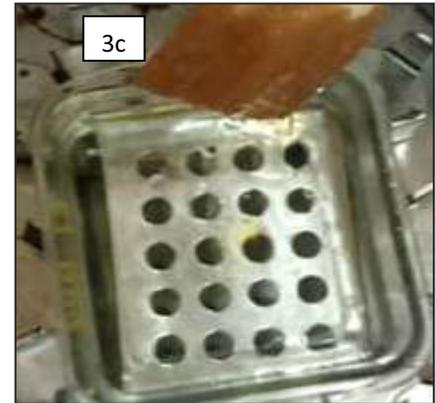


Fig.3c Oil absorbed sorbent

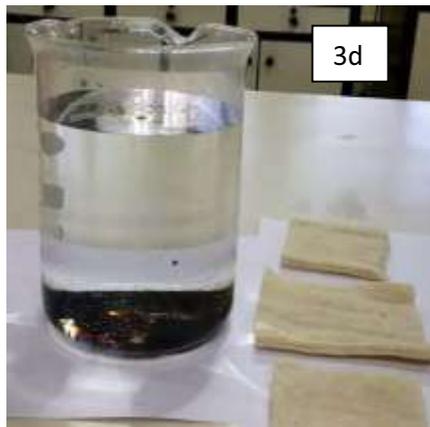


Fig.3d Oil in underwater

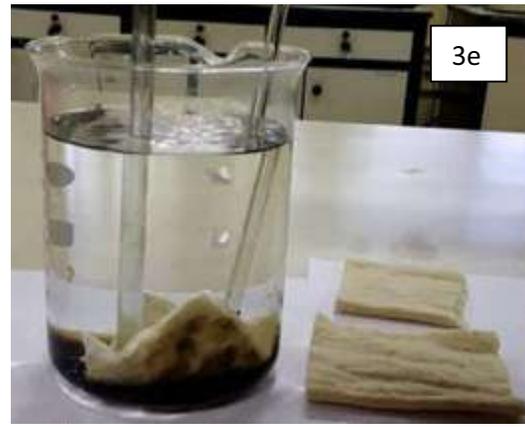


Fig 3e Photograph of underwater oil spill clean-up

Table-1 Absorption properties of sorbent in motor oil and water

Cotton waste-based sorbent	Absorption capacity of Oil (g/g) under static condition	Absorption capacity of oil in Water (g/g) under dynamic condition	Absorption capacity of Water (g/g) under Dynamic conditions
Polypropylene matt	12.3	12.7	0.1
Polyurethane matt	10.9	14.2	1.7
Carding flat stripping	30.3	29.1	0.01
Carding flat stripping lint	30.0	28.2	0.09
Blow room black dropping lint	22.0	20.6	0.18
Blow room super dropping	25.9	27.2	0.15
Blow room super dropping lint	21.3	20.2	0.1

Table-2 Absorption properties of sorbent in diesel oil and water

Cotton waste-based sorbent	Absorption capacity of Oil (g/g) under static condition	Absorption capacity of oil in Water (g/g) under dynamic condition	Absorption capacity of Water (g/g) under Dynamic condition
Polypropylene matt	6.1	8.5	0.1
Polyurethane matt	4.7	7.2	1.3
Carding flat stripping	12.5	13.1	0.1
Carding flat stripping lint	10.2	12.7	0.18
Blow room black dropping lint	9.8	10.2	0.1
Blow room super dropping	11.5	12.1	0.12
Blow room super dropping lint	11.3	11.5	0.07

Fig 3a, 3b, 3c, 3d, and 3e are the photographs of oil spill clean-up by using chemically modified sorbent in surface water and underwater. Fig.3a is the photograph of floating oil on the surface of the water. Fig 3b and 3c are the photographs of oil removal and oil-soaked sorbents respectively. Fig.3d and 3e are the photographs of oil underwater and its clean-up respectively. It was observed that chemically modified cotton absorbs only oil and repels water which is in line with the requirements of a good oil sorbent. In short oil sorbents designed for the oil spill, and clean-up in water bodies should exhibit the property of super oleophilicity and super hydrophobicity. Super hydrophobic fabric cannot be wetted by immersing in water as they always tend to float on the surface of the water. When a hydrophobic fabric was forcibly immersed underwater, a silver colour reflection appeared on the fabric surface due to the reflection of light by the air bubbles trapped on the fabric surface, and this is reported as the plastron layer formation [9]. This further highlights the super hydrophobic effect of oil sorbents. Oil, having high density settles in the bottom of water during oil spillage as shown in figs 3d & 3e. If we forcefully push a super oleophilic and super hydrophobic sorbent into the bottom of the water, the sorbent will absorb only oil. The image clearly shows that the entire oil was absorbed by the sorbent, even in water.

3.2 Water repellent property of sorbent

The water repellent property of sorbent was further characterized by contact angle measurement. The contact edge was the edge; routinely estimated through the liquid-vapours interface that meets a strong surface.

For the most part, if the water contact edge is lower than 90° the surface is viewed as hydrophilic, and if the water contact edge is higher than 90° the surface is considered hydrophobic, whereas, if the water contact angle is greater than 150° the solid surface is considered super hydrophobic [10]. This is shown in Fig 4b. Fig 4a and Fig 4b are the images of the contact angle of untreated and chemically modified sorbents. Fig 4c is the photograph of coloured water drops on the surface of the chemically modified sorbent.

The contact angle of the chemically modified cotton and control cotton was measured with an $8\ \mu\text{l}$ deionized water droplet (Easy drop analyzer D-5A 100 of Kruss with Stringray camera) instrument at room temperature. The entire contact angle was determined by averaging the values obtained at 5-6 different points on each sample surface.

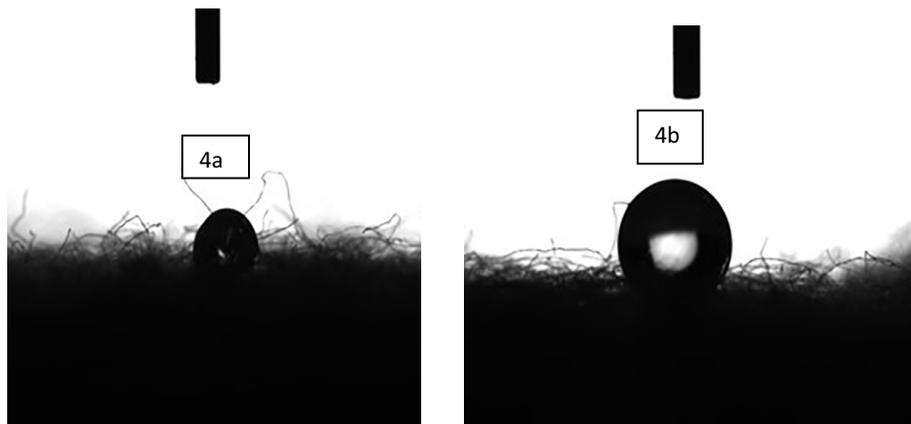


Fig. 4a & 4 b Image of contact angle of untreated and chemically modified sorbent.

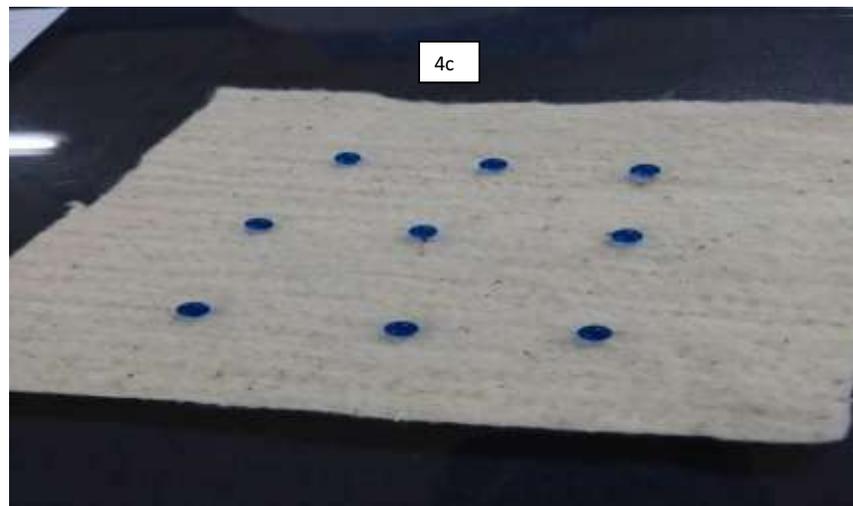


Fig.4c Photograph of coloured water drops on the surface of the chemically modified cotton sorbent.

The images Fig.4a & 4b indicate that the hydrophobicity of cotton increases by treating it with chemicals as the contact angles enhanced from 90° to 155° . There are numerous miniaturized scale convexes circulated on the outside of the film, the smaller scale convexes comprise many sub-micrometer pores and nanoparticles which take after the exemplary geology of a lotus leaf. In this way, the synergistic impact of various leveled unpleasantness from nano – small scale double structure of SiO_2 particles and hydrophobic/oleophilic nature of $-\text{CH}_3$ bunch brings about the super-hydrophobicity and Super oleophilicity of the treated surface [10]. The expansion in water repellence of a regarded texture because of the nearness of the nanoparticles on the textured surface can be credited to the expanded unpleasantness on a superficial level. The wettability of the surfaces with the fluids is controlled by the synthetic structure as well as by the geometry of the surface.

Further, we have tested the chemically modified sorbent as per the AATCC-22, water repellency spray test. The tested specimen was comparable with AATCC standard photographs of rating 100 (No sticking or wetting of upper surface). This further supports the super hydrophobic property of sorbent.

3.3 Morphology of nanoparticle on the sorbent

The morphology of synthesized silica nanoparticles and chemically modified cotton sorbent was studied by JSM IT 200 JOEL Scanning Electron Microscope.

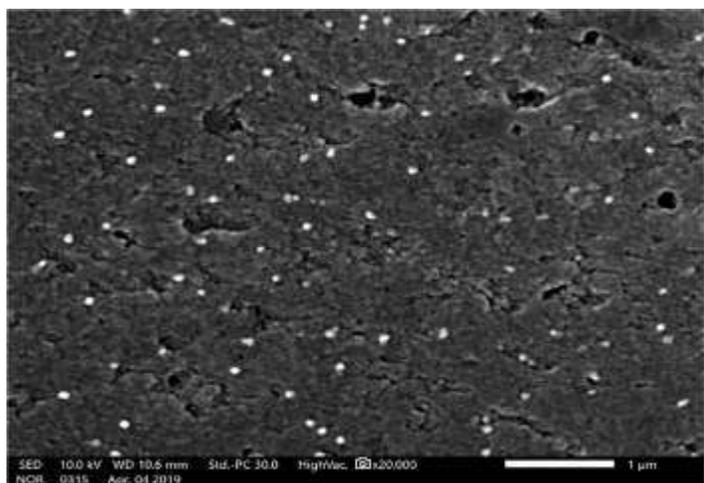


Fig 5 SEM Micrograph of nanoparticle

SEM micrograph of synthesized SiO_2 sol-gel is shown in Fig.5. When measured, the size of silica nano- particles was in the range of 70-100 nm and spherical. The size distribution varies between a diameter of 70 to 130 nm and shows the particles are almost uniformly dispersed.

Fig 6a, 6b, 6c, and 6d are the topographical changes of cotton viewed under the Scanning Electron Microscope. Fig.6a is the SEM micrograph of raw cotton, Fig. 6b is the SEM micrograph of pre-treated cotton, Fig.6c is the SEM micrograph of sol-gel treated cotton, and Fig.6d is the sol-gel

and HDTMS treated cotton. Fig 6b image shows that the Pre-treating of cotton with HNO_3 imparts many subtle textures and wrinkles on the fabric surface, which exposed more hydroxyl ions to attach with nanoparticles. Fig 6c image shows the fabric surface covered with nanoparticles. In fig-6d, it can be seen that after chemical modification of cotton with sol-gel and HDTMS, SiO_2 particles attached to the surface evenly. These nanoparticles, impart micro roughness and thus enhance the water repellent property of sorbent.

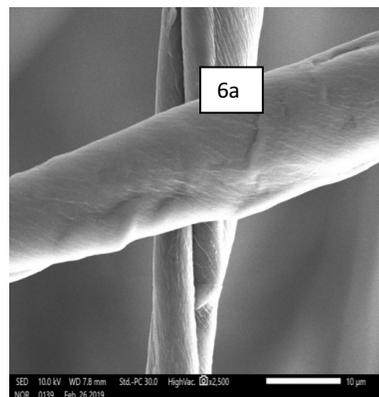


Fig.6a- SEM Micro graph of raw cotton

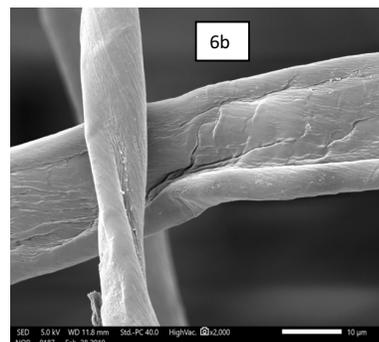


Fig.6b SEM Micro graph of pre-treated cotton

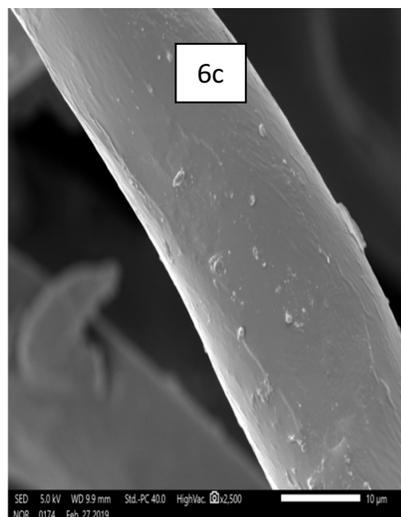


Fig.6c SEM Micro graph of nano Particle-treated cotton.

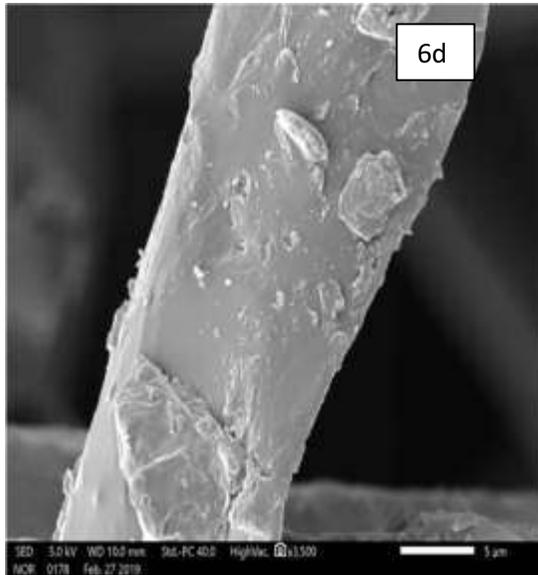


Fig.6d SEM Micro graph of chemically modified cotton

3.4. Bio- degradability of chemically modified sorbent

Soil burial test was carried out on a laboratory scale to examine the bio-degradability of chemically modified cotton sorbent and oil soaked cotton and the same was compared against polypropylene sorbent. Five test specimens of each sorbent with a dimension of 50 cm² in size were buried in the garden soil at a depth of 1-2 feet. The soil was kept moist with water. Specimens were kept under the soil for 90 days. After 90 days period, the specimens were retrieved from under the soil pit. It was found that chemically modified cotton and oil soaked cotton completely degraded and fully disintegrated in the soil whereas polypropylene sorbent was intact without any degradation. It indicates that cotton, even if chemically modified is fully biodegradable as add on nanoparticles are very less which will not affect the biodegradability. Oil soaked cotton sorbent was also found to be fully biodegradable. Fig7-9 is the photographs of oil sorbents under various stages of biodegradation.



Fig.7 Sorbents with a label before soil burial test

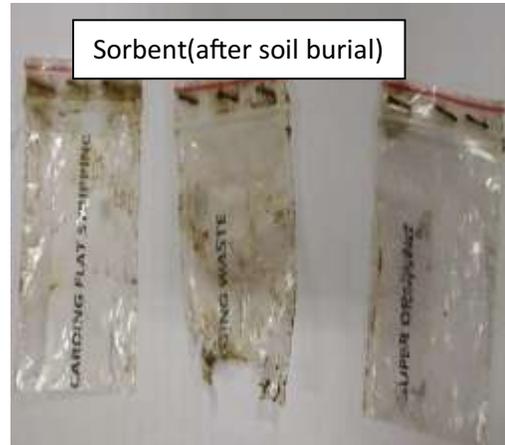


Fig.8 photograph after soil burial test



Fig.9 photograph of polypropylene before and after soil burial test

Fig. 7 is the photographs of sorbents before the soil burial test. Fig. 8 is the photograph after 90 days of the soil burial test, where we can see only labels as the sorbents have completely degraded. Fig.9 is the photograph of polypropylene sorbent before and after the oil burial test. From fig.9 it is clear that after being subjected to 90 days of soil burial test polypropylene sorbent is intact and not degraded.

3.5. Oil recovery and reusability of sorbents

The most measure for judging the reusability of the oil sorbent is the number of cycles it can endure without getting unusable due to tearing, smashing, pulverizing, or other common deterioration. Other components are the rate of diminishing in its oil absorption capacity and the rate of oil that can be outset with a sensible effort and equipment.

To assess the oil recuperation and reusability of sorbent, counterfeit sea water (500ml) was placed in a 1000 ml tray and 50g of motor oil was added to it. 4g of the sorbent material was then placed in the tray and shaken in a laboratory shaker at a frequency of 110 cycles/min for 15 min. The sorbent with the ingested oil was weighed and then squeezed between the two rollers at a pressure of 2-5N/cm. After evacuating the oil, the sorbent was re-weighed to determine the oil recuperation. The squeezed sorbent was once more utilized in the sorption process as before. The efficiency of sorbent and reusability was determined by oil sorption capacity each time after repeated sorption and desorption cycles.

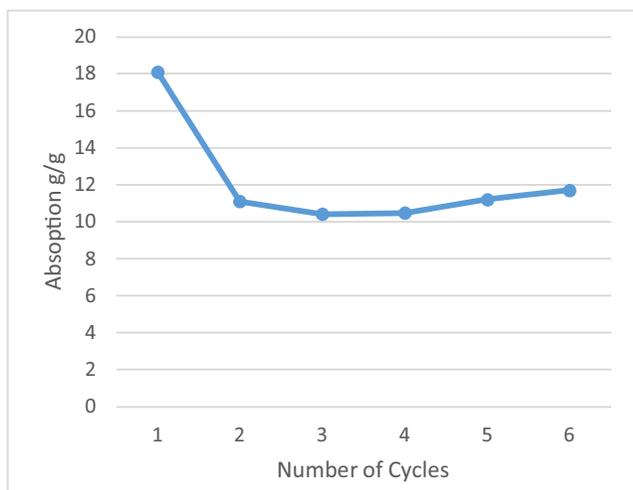


Fig-10 Oil recovery and reusability of sorbent

Fig 10 depicts a graph of absorption of sorbent (g/g) versus the repeated use cycle of sorbent. When a fresh sorbent was put in an oil bath it can absorb 18g/g of oil from an oil water media. After extrusion of the oil, the same sorbent could take up 11g/g oil in the 2nd absorption cycle. Further 3rd 4th and 5th cycles the retention capacity of the sorbent was nearly the same. During the primary six cycles of sorbent recycling, the rate of decline in the sorbent was greatest, with the most significant reduction occurring during the first interaction. This happens because a few oils were still displayed on the sorbent surface amid the extrusion process. In addition, cavities exist inside the cotton and it is not simple to discharge these after oil absorptions. During the repeated cycling utilize, part of the non-woven fabric structure distorts and thereby reduces the interstitial space.

References

- 1] Annunciado TR, Sydenstricker THD & Amico S C, Marine pollution Bull,50(2005)1340.
- 2] Wei Q F, Mather R, R Fortheringham AF & yang R D, Marine pollution Bull,46(2003) 780.
- 3] Denizceylan S& Burakkaracik,S Environ Sic Technol,43(2009)3846.
- 4] S.E Allen, B.W Smith, K.A Anderson; Impact of the deep water horizon oil spill on bioavailable polycyclic aromatic hydrocarbons in Gulf of Mexico coastal waters, Environ.Sci.Technology.46(2012), 2023-2039.
- 5] R.J Seymour, R.A Geyer; Fates and effects of the oil spill, Annu.Rev.Energy Environ. 17(1992) 261-283.
- 6] Q. F Wei, R.R Mather & A.F Fotheringham ; Oil Gas Science Technology, 62(2005),407
- 7] M. Hussein, A.A Amer, A.El-Maghraby & N.A Taha; Int J. Environ Sci Technol, 6(1) (2009) 123
- 8] M. Radetic, V.Ilic, D.Radojevic, R.Miladinovic, D.Jocie, and R. Jovanic; Efficiency of recycled wood-based non-woven material for the removal of oil from water, Chemosphere, vol. 70, Jan (2008) pp 525-530.
- 9] M.E Yazdanshenas, M.Shateri-Hkalilabad, One-step synthesis of super hydrophobic coating on cotton fabric by ultrasound irradiation, ind.Eng.Chem.Res.52(2013) 12846-12854.
- 10] Gui-long Xu, Li Li Deng, Pi-Hu-Pi, Xiu-Fang Wen, Da-Feng Zhen, Zhi-Qi Cai, Qiang Chen & Zhu-Ru Yang; Preparation and characterization of superhydrophobic/superoleophilic SiO₂ film, (2011) 127:1,9-14.

Consequently, the rate of absorption of oil reduces. However, after six times reuse, the adsorption rate of cotton was approximately 60%.

4.0 Conclusion

It has been seen that there is an extraordinary potential for oil sorbents produced using characteristic strands to supplant engineered oil sorbents. Thus, in this paper, a novel technique dependent on the synthetic adjustment of waste cotton fibre utilizing nanoparticles has been effectively evolved. Notwithstanding this has given evidence of the warm trustworthiness which could be practiced by the circuit of silica nano molecule onto the cotton the nearness of nanoparticles and nano roughening impact which could be made by nanoparticles statement was seen from SEM pictures. The very hydrophobic property of the sorbent was obvious through the contact angle which was more than 150°. The created sorbent was seen as too hydrophobic and too oleophilic in nature. The oil absorption limit of the created sorbent was a lot higher than financially accessible polypropylene sorbents. The sorbent can be reused in any event 5-6 times without debasement. The created item can be utilized as oil sorbent to clean oil spillage ashore, water surface, and submerged. The sorbent was seen as completely biodegradable. Using a waste material to a worth included, the biodegradable item was a significant accomplishment in this exploration workout.

Acknowledgment

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Evaluation of Geogrid as Reinforcement in Soil Layers using Light Weight Deflectometer and Field CBR Test



Sunil Dighe, Lekhaz Devulapallib*

The Bombay Textile Research Association, L B S Marg, Ghatkopar (W), Mumbai 400086

Abstract

The inclusion of the geosynthetics as reinforcement in the pavement layer improves the performance of pavement during its service life. In the present investigation, geogrid is added as reinforcement in compacted layer of soil. The long-term performance of the pavement majorly depends on proper compaction of the pavement layers and reinforced material used in it. In this paper modulus and strength of the geogrid reinforced and unreinforced soil layers is investigated by using Light Weight Deflectometer (LWD) device and field California Bearing Ratio (CBR) test apparatus respectively. The LWD is the non-destructive, portable testing device used to evaluate the modulus and deflection. While the field CBR test is conducted to evaluate the strength of the geogrid reinforced and unreinforced soil material in the test tank. The layer of geogrid (750 x 450 mm) is placed on first compacted soil layer as reinforcement. Over that 100 mm thick soil material is placed and compacted to required density. The LWD and field CBR tests are conducted on top surface of compacted soil material in unreinforced as well as reinforced test tank. Based on the results it is concluded that geogrid had improved the modulus and strength of the compacted soil materials by 31.70% and 29.84% respectively in comparison to unreinforced soil material.

Keywords:

Light weight deflectometer; Field CBR; Geosynthetic; Geogrid

Citation

Sunil Dighe, Lekhaz Devulapallib - "Evaluation of Geogrid as Reinforcement in Soil Layers using Light Weight Deflectometer and Field CBR Test", *BTRA Scan* - Vol. LI No. 4 Oct. 2022, Page no. 9 to 13

1.0 Introduction:

In civil engineering practice over the recent decades the use of geosynthetic as reinforcement during pavement construction becomes very popular due to its potential benefits. The use of geosynthetics as pavement reinforcement improves bearing capacity substantially. The bearing capacity of the pavement is a major concern at weak subgrade region and also quantity of the soil fill available during construction is limited in most part of the country. The use of geosynthetics as pavement reinforcement solves this problem by increasing soil bearing capacity and increases material confinement in pavement layers (resulting in a reduction in pavement design thickness). In the present investigation geogrid is used as reinforcement in compacted layer of soil. The use of geogrid as reinforcement in the

pavement layer, increases interlocking between soil particles, restricts lateral movement, and reduces vertical subgrade deformations. These geogrid reinforcement properties in the compacted layer soil are measured by using LWD [1]. Geogrid reinforcement improves the load bearing capacity of the pavement with a single layer of reinforcement. This increase in pavement bearing capacity is checked by determining the modulus and deflection of the pavement using LWD [2], [3]. The CBR value of the pavement layers determines the pavement's long-term performance. The use of geogrid in pavement layers improves the CBR value significantly when compared to unreinforced sections, and it is measured using a field CBR test that is widely accepted in the construction field [6], [7]. In this paper, soil material is compacted in layers in the test tank to required density. During the compaction of the soil material, single layer of geogrid are placed at particular

*Corresponding author,

E-mail: soillab@btraindia.com

heights in the test tank. The LWD and field CBR tests are then conducted as per planned sequence in reinforced and unreinforced test tank. Based on results the strength and modulus values are used to evaluate the performance of the reinforced soil material in comparison to the unreinforced soil is discussed.

2.0 Materials

The soil and geogrids used in the investigation are depicted in Fig.1-2. The engineering properties of these materials are determined in the laboratory by conducting various tests according to IS codes. The results of soil and geogrid are tested in laboratory and listed in table1 and table 2 below.

Table 1: Engineering properties of soil material

Sr.no	Type of Material	Test Parameter	Value	Reference Code
1	 <p><i>Fig. 1 soil material</i></p>	Water content	15.25 %	IS:2720 (part-2) [8]
		Specific gravity	2.31	IS: 2720 (part-3/ sec2) [9]
		Soil gradation	Well graded sand with little fines (SW)	IS: 2720 (part-4) [10]
		Plasticity index	Non plastic	IS: 2720 (part-5) [11]
		Dry density	1.78 g/cc	IS: 2720 (part-7) [12]
		CBR unsoaked	4.10 %	IS: 2720 (part-16) [13]
		CBR soaked	3.24 %	

Table 2: Engineering properties of geogrid material

Sr. No	Type of material	Test Parameter	Value	Reference code
1	<p>Geo grid</p>  <p><i>Fig. 2 Geogrid material</i></p>	Nominal Tensile Strength	60 kN/m	ASTM: D6637 [15]

3.0 Test Apparatus Description and Testing Procedure

3.1. Field California Bearing Ratio

Field CBR test apparatus is used in this paper to determine the California Bearing Ratio (CBR) of the soil, and a typical image of the test apparatus is shown in Fig.3 (A). The entire field CBR test apparatus is assembled, and the entire assembly is secured to the fix support is shown in Fig 3. (B).

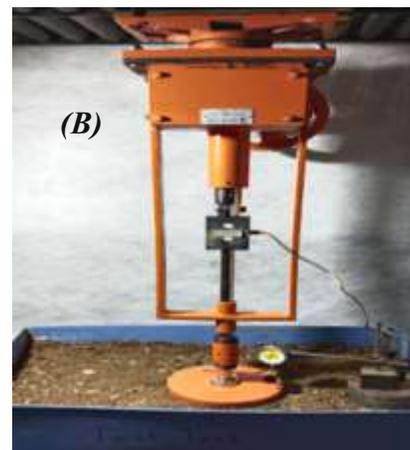
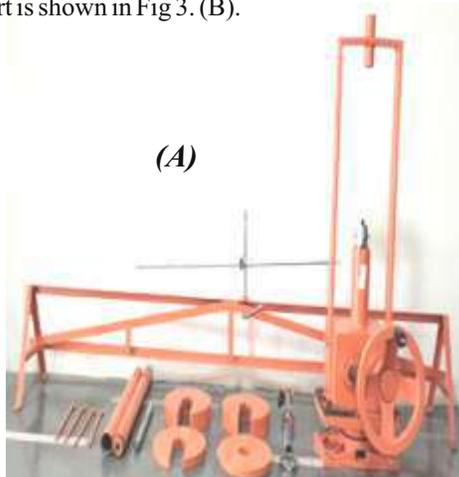


Fig.3. (A) Typical Field CBR apparatus(B)Field setup of field CBR test apparatus

The Field CBR test procedure consists following steps. The test surface area is cleaned and levelled. The equipment is secured to fixed support to provide load reaction. The surcharge annular weight of 5 kg is kept in position on the surface to be tested. The piston is lowered through hole of annular weight to establish full contact between test surface and piston by applying smallest load (not exceeding a total load of 40 N). While the seating load is on the piston, a 3 to 6 mm layer of clean sand is spread over the surface to be covered by the surcharge annular weight so that load distribution is uniform. The proving ring and dial gauge reading is set to zero before application of load on piston. Then the load is applied on piston through wheel arrangement of the equipment. The penetration rate of the piston is kept approximately 1.25 mm/min. The applied load is recorded at the penetration value equal to 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 4.0, 5.0, 7.5, 10.0 and 12.5 mm. The maximum load and corresponding penetration value are also noted if it occurs before the penetration value is less than 12.5 mm [14].

3.2 Light weight deflectometer

In this paper, Dynatest LWD is used to estimate the modulus of compacted soil layers, and it consists of the elements shown in Fig.4. The LWD procedure consists of the following steps. Initially, the test site is levelled, and ribbed rubber is placed on the levelled surface. The LWD is then kept over ribbed rubber, along with the loading plate. To ensure good contact between the loading plate and the soil, the three initial drops are released from a predetermined height. Then next three drops are used to measure deflection and modulus. The LWD estimate the modulus value from measured a deflection (generated after the impact) based on the force needed to generate a given deflection for that soil type [16], [17].

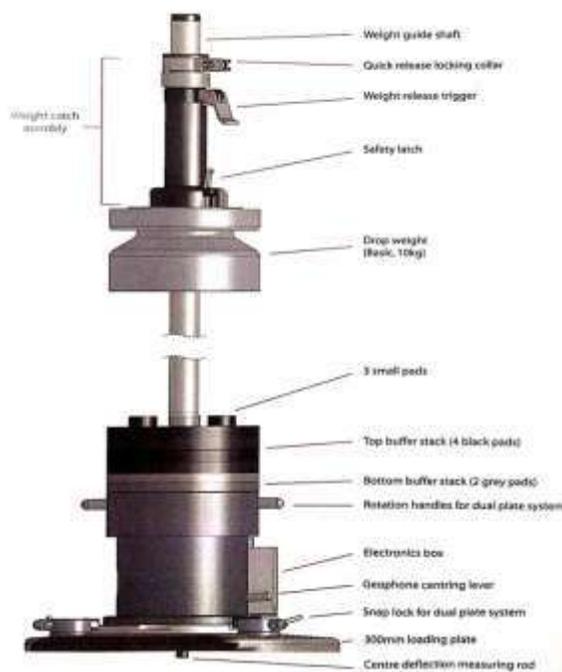


Fig.4 Typical Image of LWD [17]

4.0 Test Sequence

The schematic cross-section of the reinforced test tank is depicted in Fig. 5.

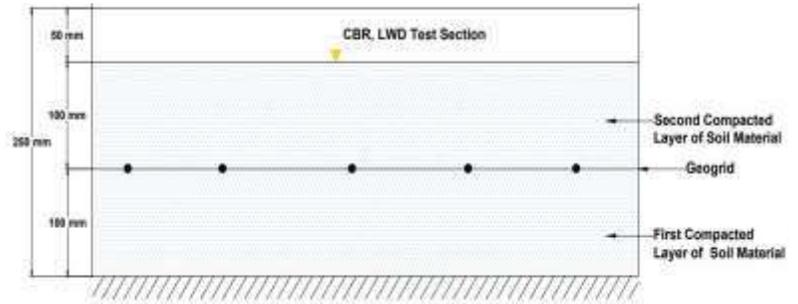


Fig 5 schematic cross-section of the reinforced test tank

The test tank 800 mm x 500 mm x 250 mm (i.e., Length, Width and depth respectively) is kept on the test location as depicted in (Fig. 6). To initiate the testing, the first layer of soil material (100 mm) is placed in tank and compacted to required density. On top of the first layer, second layer 100 mm thick soil is compacted to required density in case of unreinforced test. While in case of reinforced test the geogrid 750 x 450 mm is placed on the first compacted layer of soil in the tank and over that 100 mm soil material is compacted to required density. The field density of compacted soil material is checked using pave tracker depicted in Fig 9. The LWD and CBR test are conducted on top surface unreinforced as well as reinforced test tank. The performance values are noted. The sequential images of the test conducted are depicted in Fig. 7-10.



Fig.6. Test tank– 800 x 500 x 250 mm

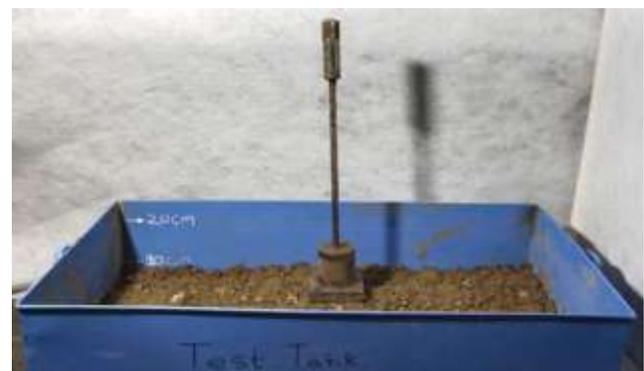


Fig7. Compaction of the 100 mm soil material



Fig 8. Placing of geogrids above 100 mm compacted soil material



Fig 9. Placing and compaction of 100 mm over geogrid and checking density by pave tracker



Fig10. Conducting LWD and Field CBR test on the reinforced test section

5.0 Results and Discussion

The performance of LWD and field CBR test on unreinforced and reinforced soil material is depicted in Fig 11-12. The test results are shown in table 3. The test results LWD and field CBR shows that modulus as well as strength values of reinforced soil increased in comparison to unreinforced soil material by 31.70% and 29.84% respectively.

Table. 3 CBR test results of the reinforced and unreinforced test section at different layers

Test Name	Test Parameter	Unreinforced soil material	Reinforced soil material
LWD	Modulus (MPa)	20.5	27
Field CBR	CBR (%)	2.58	3.35

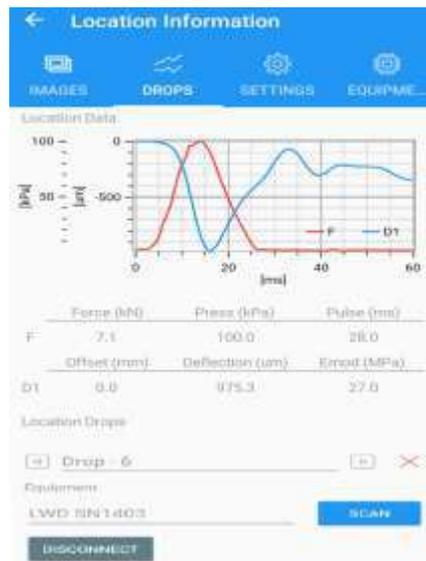


Fig 11. LWD test results of unreinforced and reinforced soil material

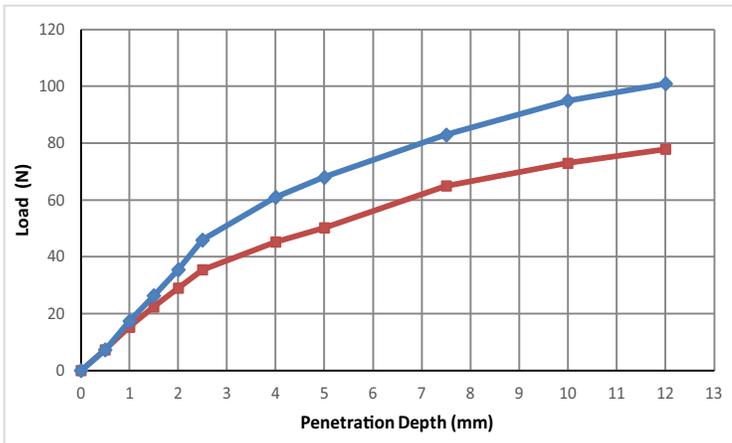


Fig: 12 Field CBR test results of reinforced (Blue line) and unreinforced soil material (dark red line)

The results show that the strength and modulus values of geogrid reinforced sections are increased than that of unreinforced sections. The use of geogrid as reinforcement in soil improved the bearing capacity of compacted layers by interlocking soil materials and provided material confinement within layers. The soil-bearing capacity increases with the addition of one layer of reinforcement. The reinforcement also provides additional support in response to the applied load, by the deformation of reinforcement in the upward direction below the region of applied load [6]. This mechanism was latter introduced by Giroud and Norway (1981) as membrane tension support. The soil material is weak in tension and geogrid is strong in tension. So as the soil is reinforced with geogrid the tensile load is taken by the geogrid. The part of the applied load is transmitted laterally to the adjacent soil at the soil geogrids interface which increases the bearing capacity of reinforced

References

- Elhakim, A.F., Elbaz, K. and Amer, M.I., 2014. The use of light weight deflectometer for in situ evaluation of sand degree of compaction. HBRC Journal, 10(3).
- Jitarekul, P., Sawangsuriya, A. and Singhatiraj, P., 2017. Integration of pavement layer evaluation using LWD for road safety control. Procedia engineering, 189, pp.111-117.
- Dwivedi, S. and Suman, S.K., 2019. Quality assessment of road shoulders using light weight deflectometer and geogauge. Int J Recent Techn Eng.
- Giroud, J.P. and Han, J., 2004. Design method for geogrid-reinforced unpaved roads. I. Development of design method. Journal of Geotechnical and Geoenvironmental Engineering, 130(8), pp.775-786.
- Bandyopadhyay, K. and Bhattacharjee, S., 2010, December. Comparative study between laboratory and field CBR by DCP and IS method. In Indian Geotechnical Conference, Kolkata, Indian, December.
- Carlos, D.M., Pinho-Lopes, M. and Lopes, M.L., 2016. Effect of geosynthetic reinforcement inclusion on the strength parameters and bearing ratio of a fine soil. Procedia Engineering, 143, pp.34-41.
- Balamaheswari, M., Anitha, B., Kanimozhi, B. and Prabhu, N., 2022. Improvement of California Bearing Ratio value in weak subgrade soil with the developed anchored geogrid. Materials Today: Proceedings, 49, pp.1537-1542.
- IS: 2720 (Part 2):1973, Methods of Test for Soils- part 2: Laboratory water content determination.
- IS: 2720 (Part 3/sec 2):1980, Methods of Test for Soils- part 3/sec2: Laboratory specific gravity of soil.
- IS: 2720 (Part 4):1985, Methods of Test for Soils- part 4: Laboratory Grain size analysis of soil.
- IS: 2720 (Part 5):1985, Methods of Test for Soils- part 5 : Laboratory determination of liquid limit and plastic limit
- IS: 2720 (Part 7):1980, Methods of Test for Soils- part 7: Laboratory determination of dry density and optimum moisture content.
- IS: 2720 (Part 16):1987, Methods of Test for Soils-Part (16): Laboratory Determination of CBR.
- IS: 2720 (Part 31):1990, Methods of Test for Soils-Part (31): Field Determination of California Bearing Ratio.
- ASTM: D6637, Standard Test Method for Determining Tensile Properties of Geogrids by the Single or Multi-Rib Tensile Method.
- ASTM E2835, 2020 Standard test method for measuring deflections using a portable impulse load device.
- Dynatest 3031 LWD test system owner's manual. <https://dynatest.com/equipment/light-weight-deflectometer/>, Accessed 12th Oct 2022. inetics of the curing process by FTIR spectroscopy," Polym. Networks Blends, vol. 7, no. 1, pp. 1–11, 1997, Accessed: Oct. 14, 2022.

soil [4]. This increase in the bearing capacity of soil can correlate to an increase in the strength and modulus value.

6.0 Conclusions

The CBR values and modulus on unreinforced and reinforced soil material is determined by performing field CBR test and LWD in the test tank. Based on the results the following broad conclusions are drawn.

- The performance of compacted soil material is greatly enhanced due to the inclusion of geogrid as reinforcement
- The field CBR values of reinforced soil material increased by 29.84 % for unreinforced soil material
- The modulus values of reinforced soil material are increased by 31.70 % with respect to unreinforced soil material

Note: The present investigation is conducted on a single source of soil and geogrid. It is needed to perform the tests on different types of soil material and geosynthetics to achieve consistency..

Future scope

In the present investigation, the LWD and Field CBR test are conducted on particular soil material. The geosynthetics material as reinforcement in soil material is complex phenomenon; hence there is a scope for further research. The research may be conducted on following aspect.

- Evaluation of geogrid as reinforcement with different type of soil material
- Evaluation of different type of geosynthetic as reinforcement with different type of soil material

Cure Kinetic Study of Unsaturated Polyester Resin

Amol G. Thite*, Deepali M. More

The Bombay Textile Research Association, L B S Marg, Ghatkopar (W), Mumbai 400086

Abstract

An unsaturated polyester resin is one of the principal matrices used in fibres reinforced plastics (FRP) products. Being a pre-polymer the cure kinetics plays an important role in making successful FRP products. Hence, this study deals with the cure kinetics of unsaturated polyester resin. It includes the optimization of gel time, curing time and temperature of it. The gel time, which indicates the formation of cross-linking structure, was studied by varying the weight percentage of the Methyl Ethyl Ketone Peroxide (MEKP) catalyst and Cobalt Octane accelerator. The optimum gel time of 15-16 minutes was found at the 1:1 wt% level of MEKP and Cobalt Octane concentration. A differential scanning calorimetry was used to obtain conversion profiles of the resin at different temperature ranges from 25–45°C. From the DSC study, the suitable curing temperature of unsaturated polyester resin was identified as 45°C. Fourier transform infrared spectroscopy was used to study the effect of time on the curing of unsaturated polyester resin. From FTIR analysis, the major conversion of resin was found at 35 minutes.

Keywords:

Cure kinetics; curing time; gel time; unsaturated polyester resin

Citation

Amol G. Thite, Deepali M. More - "Cure kinetic study of unsaturated polyester resin", *BTRA Scan* - Vol. LI No. 4 Oct. 2022, Page no. 14 to 18

1.0 Introduction

Composites are composed of resins, reinforcements, fillers, and additives. Each of these constituent materials or ingredients plays an important role in the processing and final performance of the product [1]. Among these, resins (matrix) are an important constituent of the composite. It is the "glue" that holds the composite together and influences the physical properties of the product [2]. Therefore, these thermoset resins are frequently used as matrix materials in polymer composites such as fibre-reinforced plastic and polymer concretes [3]. The most common thermosetting resin used in the composites industry is unsaturated polyester (USPET). The USPET resins are known as the workhorse of the composites industry as they represent approximately 75% of the total resins used and possess good mechanical properties, excellent thermal stability and weather resistance. Therefore, they are the most versatile, globally used, thermoset polymer [4], [5].

Chemically, USPET resins are nothing but macromolecules with a polyester backbone and belong to the category of thermoset resin. They are broadly defined as condensation products of organic dicarboxylic acids and di-functional alcohols (glycols) [6]. It contains an unsaturated material, such as maleic anhydride or fumaric acid, as part of the dicarboxylic acid component [7]. It is often blended with

vinyl group molecules like styrene (35- 45 wt%) into a reactive resin solution. This styrene acts both as a cross-linking agent and as a viscosity reducer [8]. In addition, during the curing process at room temperature, generally, organic peroxides like methyl ethyl ketone peroxide (MEKP) are mostly used as catalysts while cobalt salt is frequently used as an accelerator to control the gelling time of the resin [8], [9]. The inhibitors such as quinones and phenol are added to react with the initiating or propagating radicals to reduce their reactivity. This gives an induction period before the resin is cured and adds to its storage life [10].

When this resin is cured, the monomer reacts with the unsaturated sites on the polymer converting it to a solid three-dimensional, rigid and hard thermoset polymer structure. However, the curing kinetics of polyester resins is very complex since a free radical chain growth polymerization reaction takes place with the stages of initiation, propagation, and termination. Further, the curing of USPET resin occurs as a combination of chemical kinetics and control by diffusion. In addition, the interaction of chemical kinetics with physical phenomena like gelling makes the curing process more complex [11], [12]. Hence, the success of producing a product by processes like hand lay; resin transfer moulding or resin spraying depends on the cure kinetics as well as the gel time. Hence, the processing of these resins requires an understanding of the polymerization

*Corresponding author,

E-mail: defectanalysis@btraindia.com

reaction kinetics during cure [9],[13]. Therefore, in this present work, the initial gel time of unsaturated polyester resin was studied by varying the wt% of catalyst (MEKP) and accelerator (Co). Then, an optimized recipe was used to study the cure kinetics of the polyester resin by DSC and FTIR spectroscopy analysis.

2. Experimental

2.1 Materials

The resin transfer moulding (RTM) grade unsaturated polyester resin ESPOLTM containing phthalic anhydride (PA), propylene glycol (PG), diethylene glycol (DEG) and maleic anhydride was procured from Satyan Polymer, Mumbai, India. The peroxy benzoate (MEKP) as a catalyst with cobalt octane (3%) as an accelerator was purchased from Empee Corporation Pvt. Ltd. Bangalore, India.

2.2 Methods

2.2.1 Gel time study

The effect of amounts of catalyst and reaction accelerator on the gel time of USPET resin was studied to understand the time, and temperature superposition required for curing. The gel time was determined by manual methods based on “by eye” evaluation of the rheological behaviour of the resin during stirring, the pattern of resin fibre drawing or adhesion to the probe.

2.2.2 Cure kinetic measurements by DSC analysis

The reaction kinetics (curing temperature) of USPET resin mixed with catalyst and reaction accelerator were measured by a DSC-30 Mettler Toledo instrument. A dynamic curing reaction scan was conducted from 25 to 300°C at a heating rate of 10°C/min in a nitrogen flow rate of 10 mL/min. The isothermal runs were carried out for 60 minutes at 25, 30, 40 and 45°C temperatures. The samples are then heated to 350°C in the non-isothermal scanning mode with a heating rate of 10°C/min to determine the residual heat of the reaction (ΔH_{res}). All the reactions were conducted in hermetically sealed aluminium sample pans. The weight of the samples taken for the study ranged between 5 to 10 mg.

2.2.3 Cure kinetic measurements by FTIR spectroscopy

The reaction kinetics (curing time) of styrene monomer and polyester vinyl groups was determined by FTIR spectrometer (Perkin Elmer 2000, USA). Before acquiring a spectrum, initially, the reactants were thoroughly mixed and then one drop of the mixture was pasted between two KBR plates and mounted on a sample holder. The FTIR spectrum for each sample was acquired after 10 scans in the range of 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1} in the absorbance mode.

3. Results and discussion

3.1 Determination of styrene content and gel time

Initially, it was necessary to determine the content of styrene present in the USPET resin. Here, the weight loss method

was used to find out the amount of styrene used in the USPET resin. For that, the purchased USPET resin was kept in an oven under vacuum at 120°C for 2 h followed by a measurement of weight loss. This procedure was continued several times until the complete evacuation of styrene took place. The styrene content in the USPET resin was calculated by Equation 1 and it was found to be 41% (wt/wt).

$$\text{Styrene content} = \frac{\text{Initial Weight} - \text{Final Weight}}{\text{Initial Weight}} \times 100 \dots \dots \dots \text{Eq. 1)}$$

After the quantification of the styrene content, the gel time (Tgel) of USPET resin for making composites was studied “by eye” evaluation of the rheological behavior of the resin during stirring, the pattern of resin fibre drawing or adhesion to the probe as mentioned in the experimental section. The effect of the differential concentration of catalyst and accelerator on the gel time was studied at 23°C \pm 3°C and RH: 50% \pm 5% atmospheric conditions. The corresponding gel time is tabulated in Table 1.

Table 1. Gel time (Tgel) of unsaturated USPET at various concentrations of catalyst and accelerator

Experiment	Cobalt Octane (wt%)	MEKP (wt%)	Gel time (Tgel)
1	2	2	3-5 min
2	1.75	1.75	7 min
3	1.5	1.5	10-11 min
4	1.25	1.25	13-14 min
5	1	1	15-16 min
6	0.75	0.75	23 min
7	0.5	0.5	36 min
8	0.25	0.5	58 min
9	0.5	0.25	Above 1 h
10	0.25	0.25	Above 1 h
11	0.1	0.1	Above 1 week

From Table 1, it was observed that as the amount of both reactants decreases, the gel time proportionately increases. The value of Tgel was approximately above one week at the lowest level (0.1, 0.1) of the catalyst and accelerator, whereas it was found to be reduced significantly to 3-5 min at the highest level (2, 2) of reactants. The increase in gel time is primarily because of less number of available free radicals during the free radical polymerization reaction of USPET resin. The crosslinking reaction of USPET is shown in Figure 1.

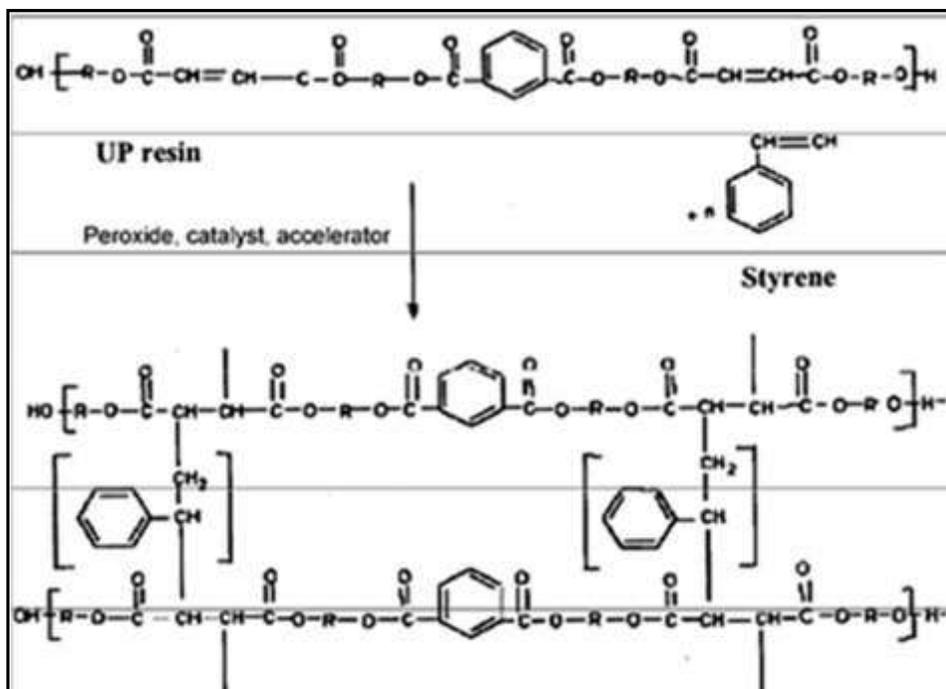


Figure 1. Cross-linking or gelling mechanism of USPET resin

During the reaction, the free radicals initiate the exothermic copolymerization reaction. Due to this, the formation of macromolecules takes place which continues to grow resulting in a gel form which is a phenomenon describing the transition of a material from a viscous liquid to an elastic solid during curing. From the chemical point of view, gelation implies the formation of a continuous network of covalent bonds in the system (i.e., the formation of a molecule with an infinitely high molecular weight). However, this transition does not occur instantaneously; rather, the viscosity of the system increases gradually, which makes it very difficult to precisely determine the gel time [11]. In addition, a certain problem is presented by the differentiation of gelation from vitrification that also leads to a solid but is not characterized by the formation of a continuous network of covalent bonds. Finally, this makes the reaction mass a semi-viscous state [11]. Further, it is also known that the lower T_{gel} can result in the formation of incomplete mould-filling defects because of the restriction of molecular chain mobility in the polymer. Whereas, a higher T_{gel} value can increase the cycle time and reduce productivity. Therefore, the value of T_{gel} should be neither too less nor too high. Hence, a (1, 1) level was selected as the optimum where the difference in gel time was not too much with the level of (2, 2).

3.2 Differential scanning calorimetry (DSC) analysis

The working temperature also has a significant role in the gel and curing time. Therefore, to understand the effect of temperature on the conversion of unsaturated polyester into gel formation during curing, a difference in heat of reaction was measured during a series of isothermal scans at 25, 30, 40, and 45°C and after it, as shown in Figure 2.

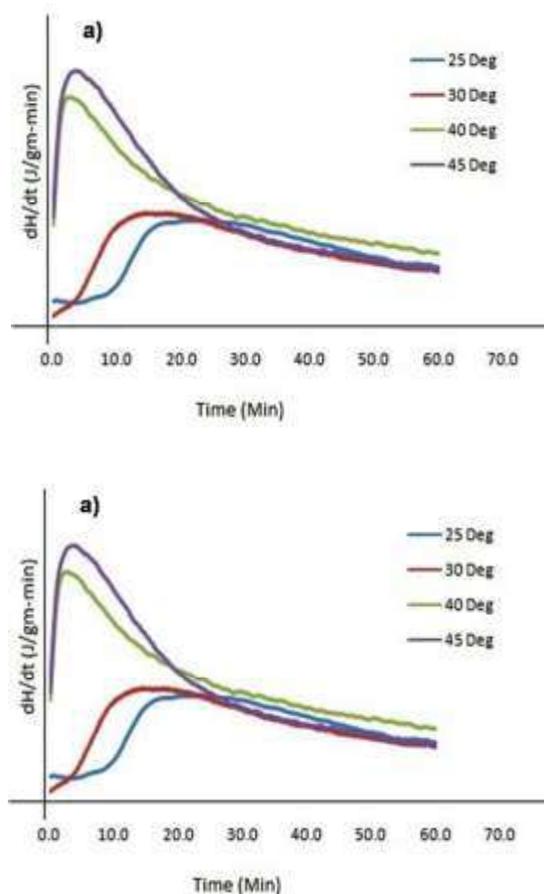


Figure 2. DSC dynamic curves obtained (a) during and (b) after isothermal scans

From Figure 2(a), it was observed that at the lower isothermal time, as isothermal temperature increases, the heat of the reaction also increases and thereafter no significant change was observed in it at higher isothermal time. In other words, the greater Tiso value, the ΔHiso value is also higher which is reflected in an increase in the conversion of the unsaturated polyester resin into the gel with an increase in reaction temperature. This is because of the styrene homo-polymerisation and its copolymerization with the C=C bond of the unsaturated polyester resin chain via the heat of the reaction involved during the isothermal period. Due to this, the molecular chains of the polymer become more mobile and the resin can further react at a higher temperature, resulting in higher final conversion and lower styrene residue. However, a total conversion was not reached at any temperature employed in this study [13]. Hence, the non-isothermal scan was carried out of the same isothermally cured sample, and a residual heat ΔHres appeared, which is the result of the reactivity of the residual functions that have not reacted during the isothermal period. The variation of this heat of reaction (ΔHres) with Tiso is shown in Figure 2(b). Therefore, the value of heat generated during the isothermal scans ΔHiso, together with the residual heat values ΔHres, are shown in Table 2.

It can be seen that the heat generated during the isothermal scan increased as the temperature increased, while the residual heat decreased. This is because of the higher mobility of double bonds and the vitrification process, which occurred at higher conversions as the temperature increased. Thus, the concentration of unreacted double bonds is reduced [14]. On the other hand, it is supposed that the quantity of (ΔHiso+ΔHres) must remain constant [15]. However, we did not find this observation, except for the greater value of Tiso. This is because of the known fact that evaporation of styrene appeared during the experiments. This leads to experimental errors in the determination of the different enthalpy values. However, 45°C was found as the optimum temperature where maximum conversion and low level of styrene residue exist.

Table 2. The heat generated during and after an isothermal scan of unsaturated polyester resin

Temperature (°C)	ΔHiso (Jg ⁻¹)	ΔHres (Jg ⁻¹)	ΔHiso + ΔHres (Jg ⁻¹)
25	99.38	173.18	272.56
30	182.43	142.69	325.12
40	260.56	66.08	326.64
45	268.86	61.83	330.69

3.3 FTIR analysis

Based on the optimum reactant concentration from gel study and temperature by DSC analysis, to optimize the curing time, the changes that occurred in the ATR-FTIR spectra at different curing times (0–35 min) were analyzed and shown in Figure 3. It has also been correlated with conversion

fraction as tabulated in Table 3. The reaction kinetics of styrene monomer and polyester vinyl groups were determined by Equations 2 and 3 in terms of changes that occurred in the characteristic peak of the C=C bond at 982 cm⁻¹ and 912 cm⁻¹ before the reaction starts (A0) and after at different times (At) for USPET and styrene respectively.

$$X_{usp\text{et}} = 1 - \frac{A_t(982)}{A_o(982)} \dots\dots\dots (Eq. 2)$$

$$X_{st} = 1 - \frac{A_t(912)}{A_o(912)} \dots\dots\dots (Eq. 3)$$

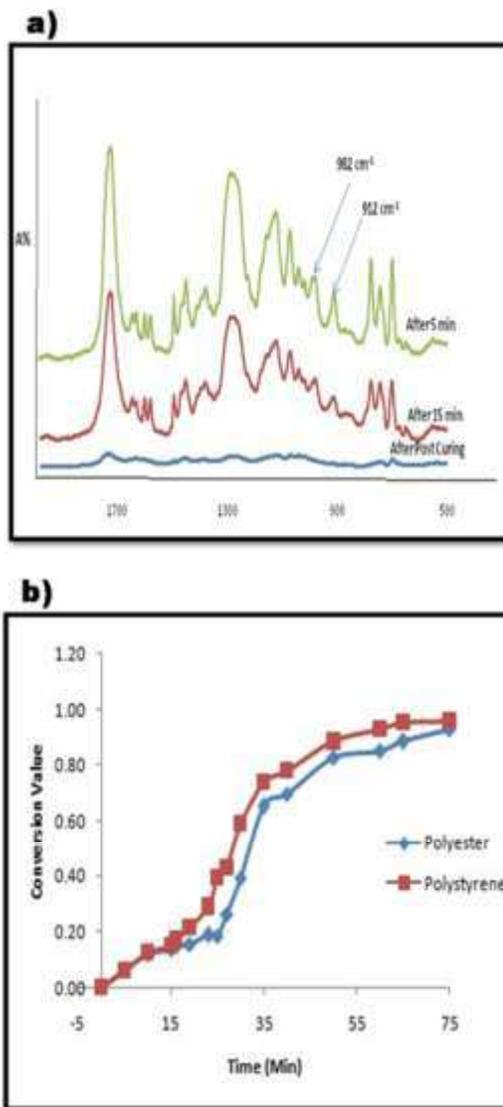


Figure 3. (a) FTIR spectra and (b) conversion fraction of unsaturated polyester resin at different curing times

From Figure 3(a), as the curing time increased, the magnitude of the characteristic peaks 982 cm⁻¹ and 912 cm⁻¹ was found to decrease. The changes obtained in the magnitude of peaks (Xup and Xst) were quantified in terms of conversion fraction and shown in Figure 3(b).

Table 3. Conversion of unsaturated polyester and styrene at different curing times

Time (min)	USPET	Styrene	USPET %	Styrene%
0	0.279	0.115	27.9	11.5
5	0.421	0.432	42.1	43.2
10	0.508	0.612	50.8	61.2
15	0.721	0.734	72.1	73.4
20	0.725	0.839	72.5	83.9
25	0.742	0.873	74.2	87.3
30	0.815	0.919	81.5	91.9
35	0.901	0.931	90.1	93.1

From Table 3, the conversion of styrene and USPET was found to be less at the beginning of the reaction. This first stage was termed before the gel point was reached. This is because, at low conversions, the cross-linking density of the reacting system would not be sufficiently high to appreciably influence the propagation mechanism of the styrene and C=C bonds of unsaturated polyester resin. At the later stages of curing, the conversion of styrene steadily increases with time, while the conversion of polyester C=C bonds slows down. This fact is due to the formation of a highly cross-linked network, so propagation reactions become diffusion controlled [16]. Hence, the mobility of small styrene molecules is less affected by the increase of cross-linking density than that of C=C units in larger polyester molecules. The major reaction was found to be completed at the curing time of 35 minutes.

References

- [1] M. Ravichandran, M. Balasubramanian, C. Anand Chairman, D. Pritima, V. Dhinakaran, and B. Stalin, "Recent developments in Polymer Matrix Composites - A review," IOP Conf. Ser. Mater. Sci. Eng., vol. 988, no. 1, 2020, doi: 10.1088/1757-899X/988/1/012096.
- [2] D. K. Rajak, D. D. Pagar, P. L. Menezes, and E. Linul, "Fiber-Reinforced Polymer Composites :," Polymers (Basel), vol. 11, no. 1667, pp. 1–37, 2019, doi: <http://dx.doi.org/10.3390/polym11101667>.
- [3] W. Li, M. Zhou, F. Liu, Y. Jiao, and Q. Wu, "Experimental Study on the Bond Performance between Fiber-Reinforced Polymer Bar and Unsaturated Polyester Resin Concrete," Adv. Civ. Eng., vol. 2021, 2021, doi: 10.1155/2021/6676494.
- [4] H. Nava, "Polyesters, Unsaturated," Kirk-Othmer Encycl. Chem. Technol., no. August, pp. 1–24, 2015, doi: 10.1002/0471238961.1615122519051212.a01.pub3.
- [5] R. Alabbas, W. Khalil, and S. Alhariry, "Synthesis and characterization of unsaturated polyester resin and studying their thermo properties," Chem. Mater. Res., vol. 9, no. 10, pp. 15–20, 2011, [Online]. Available: <https://www.iiste.org/Journals/index.php/CMR/article/view/39077>.
- [6] Bharat Dholakiya, "Unsaturated polyester resin for speciality applications," in Polyester, vol. 7, 2012, pp. 167–202.
- [7] I. H. Updegraff, "Unsaturated Polyester Resins," Handb. Compos., pp. 17–37, 1982, doi: 10.1007/978-1-4615-7139-1_2.
- [8] E. M. S. Sancheza and M. I. Felisbertib, "polymer," vol. 41, pp. 765–769, 2000, doi: DOI: 10.1007/978-94-011-5862-6_23.
- [9] S. Waigaonkar, B. J. C. Babu, and A. Rajput, "Curing studies of unsaturated polyester resin used in FRP products," Indian J. Eng. Mater. Sci., vol. 18, no. 1, pp. 31–39, 2011.
- [10] W. E. Cass and R. E. Burnett, "Inhibition of Unsaturated Polyesters," Ind. Eng. Chem., vol. 46, no. 8, pp. 1619–1624, Aug. 2002, doi: 10.1021/IE50536A032.
- [11] R. R. P. Kuppasamy and S. Neogi, "Influence of curing agents on gelation and exotherm behaviour of an unsaturated polyester resin," Bull. Mater. Sci., vol. 36, no. 7, pp. 1217–1224, 2013, doi: 10.1007/s12034-013-0591-8.
- [12] M. Wozakowska, "Kinetics of the curing reaction of unsaturated polyester resins catalyzed with new initiators and a promoter," J. Appl. Polym. Sci., vol. 102, no. 2, pp. 1870–1876, Oct. 2006, doi: 10.1002/APP.24155.
- [13] M. Avella, E. Martuscelli, and M. Mazzola, "Kinetic study of the cure reaction of unsaturated polyester resins," J. Therm. Anal. 1985 306, vol. 30, no. 6, pp. 1359–1366, Nov. 1985, doi: 10.1007/BF01914307.
- [14] J. L. Vilas, J. M. Laza, M. T. Garay, M. Rodríguez, and L. M. León, "Unsaturated polyester resins cure: kinetic, rheologic, and mechanical-dynamical analysis. I. Cure kinetics by DSC and TSR," J. Appl. Polym. Sci., vol. 79, no. 3, pp. 447–457, 2001, doi: 10.1002/1097-4628(20010118)79:3<447::AID-APP70>3.0.CO;2-M.
- [15] J. M. Salla and X. Ramis, "Comparative study of the cure kinetics of an unsaturated polyester resin using different procedures," Polym. Eng. & Sci., vol. 36, no. 6, pp. 835–851, 1996, Accessed: Oct. 14, 2022. [Online]. Available: https://www.academia.edu/18060789/Comparative_study_of_the_cure_kinetics_of_an_unsaturated_polyester_resin_using_different_procedures.

4. Conclusions

The cure kinetics of USPET resin was studied in terms of gel time, curing temperature and curing time. The gel time study shows that (1, 1) wt % of MEKP and Co-octane in unsaturated polyester resin is favourable for the preparation of composites on the RTM machines. The variation with the curing temperature (Tiso) and the heat of reaction (ΔH_{iso}) characterizing the styrene homo-polymerisation and its copolymerization with the polyester chains were determined from isothermal DSC investigation, while the residual heat of reaction (ΔH_{res}) was revealed from non-isothermal measurements. The optimum curing temperature of USPET resin was found as 45°C. From FTIR investigation the conversion C=C bonds of polyester and styrene are determined and it shows that initially, the conversion was less while in a later stage the conversion of styrene C=C bonds increases. The optimum conversion of both reactants was found at a curing time of 35 minutes.

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Declaration of conflicting interests

The author(s) declared no potential conflicts of interest concerning the research, authorship, and/or publication of this article.

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by Shri. Upendra P. Singh, Textile Secretary, GOI along with Ms Roop Rashi, Textile Commissioner, GOI and S. K. Saraf, Chairman, BTRA Governing Council. Other prominent dignitaries Mr V. K. Kohli, Jt. Textile Commissioner, Mr S. P. Varma, Addl. Textile Commissioner, P. K. Agarwal, CMD, CCI and Mr Ajit B. Chavan, Secretary, Textile Committee also graced the occasion. During the interaction, Shri. Upendra P. Singh, Textile Secretary & Ms Roop Rashi, Textile Commissioner, GOI congratulated and appreciated BTRA's initiative in developing these facilities.

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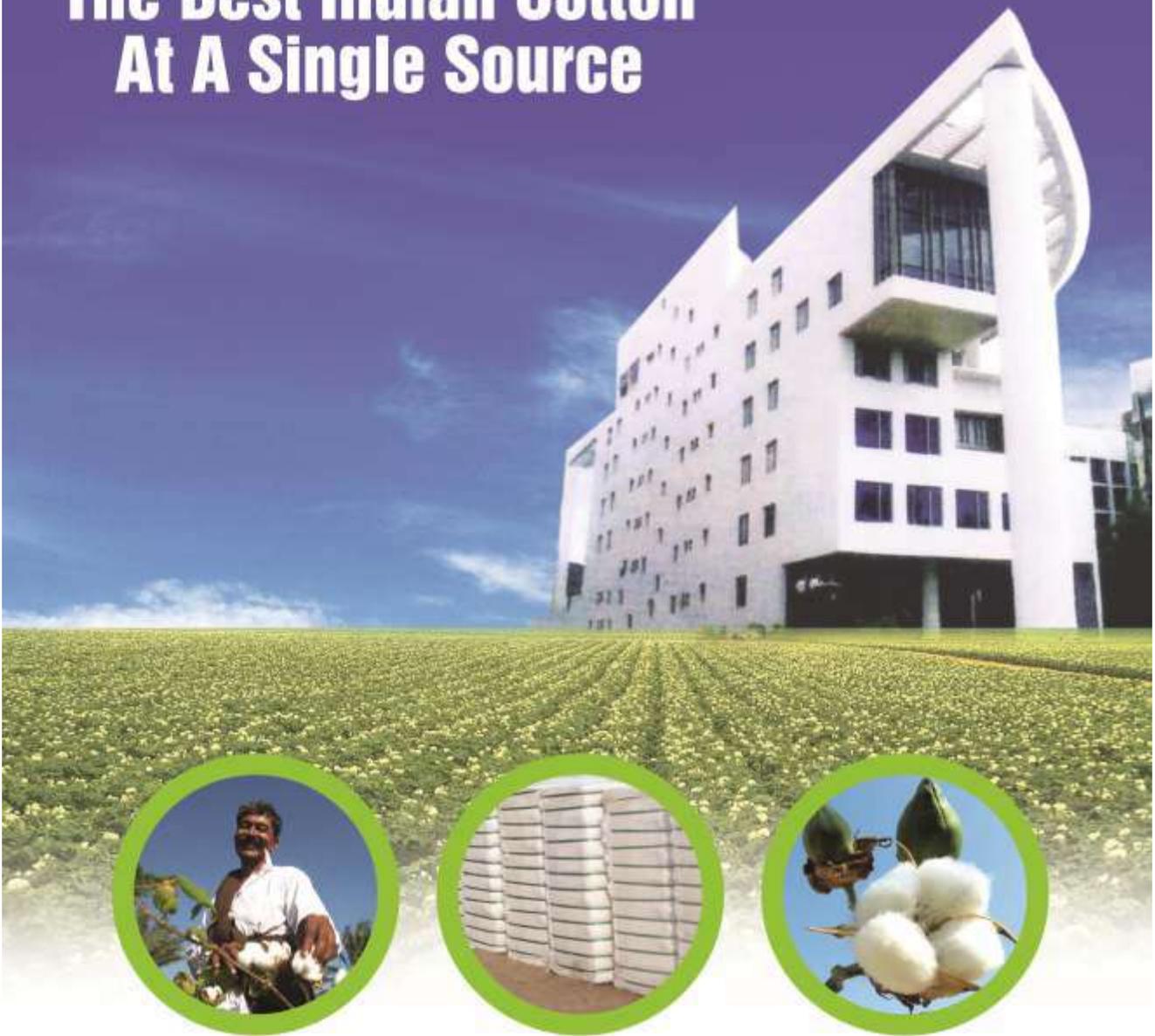
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