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Fabrication and Analysis of EPDM-Buffing Dust Composite

Manikandan G, Srinath R, Ayush Jaiswal, Akshay Naramshettiwar, Sushant Malik

Abstract*: *Wastes generated during the tanning process can be categorized as buffing wastes, shaving wastes, keratin waste (mainly from the nails and hair) and skin trimmings. The main component of all these wastes is protein.in fact keratin is one type of protein. Leather waste from chrome tanned leather, which is proteinous, impregnated with chromium, synthetic fat, oil, tanning agents and dye chemicals is one of the difficult tannery wastes to manage. In this project, various leather- polymer-EPDM composites have been made and their properties have been analyzed. polymer-leather composite modified with filler material has showed good performance with high decomposition temperature, good bonding between the constituents and low density (Hence, low weight). The strong mechanical phase interaction brought by the chemical bonding with leather interface was confirmed by FTIR analysis. Thermal Gravimetric Analysis revealed the higher decomposition temperature for the composites.

Keywords: Composites, EPDM, Glacier, Leather waste

I. INTRODUCTION

It is well accepted throughout the scientific fraternity that construction materials such as ice has its own barriers as far as its usage is concerned. Ice is regarded as a poor material for construction because of its mechanical properties such as extreme creep behavior in contrast to other normal building materials, for instance concrete. Even though ice has good strength its mechanical properties are highly dependent on temperature. Hence, it must protect from melting even though the temp. is low. The exact location of Siachen glacier is Himalayan Mountains in the Ladakh region. This place has been a major battleground between Pakistan and India for the past 60 years. The place was first reached in 1819 by W. Moorcroft and then it was explored by F. N. Workman in the year 1848 and it was inhabited by his wife for a long period of time and she gave the name "Siachen" to the glacier. The glacier upon melting flows into tributary Nubra River which in turn flows into Indus River.

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***** Correspondence Author
Manikandan G*, Department of Mechanical, SRM Institute of Science and Technology, Chennai, India. Email: xyz1@blueeyesintlligence.org
Srinath R, Department of Mechanical, SRM Institute of Science and Technology, Chennai, India. Email: nathsri464@gmail.com
Akshay Naramshettiwar, Department of Mechanical, SRM Institute of Science and Technology, Chennai, India. Email: akshaynaramshettiwar143@gmail.com
Ayush Jaiswal, Mechanical Department, SRM Institute of Science and Technology, Chennai, India.
Sushant Malik, Mechanical Department, SRM Institute of Science and Technology, Chennai, India.

The decline/retreat of the glacier is a striking evidence of the climatic changes in the regions surrounding glacier due to anthropogenic activities.

The movement of combat vehicles and other activities carried out by both the armies such as construction of buildings is the main reason for the destruction of the glacier. The information reported during the period of 1991-04 indicates an increase in temperature of 4ºc. The increase of temperature since 2004 has been exponential which has resulted in the retreat of the glacier. The deterioration estimates calculated by remote sensing facilities show that Siachen Glacier has depleted by 1.9km in longitudinal extent from 1989 to 2006. The environmental damage caused by the pollution due to tanning is immense because of the processes involved such as Deliming and dehairing. For every 1000kg of skin used 80m³ of stinking wastewater with high levels of chromium, sulfide and fats is produced. Insecticides which are used to preserve during transport also adds to the pollution of water bodies. Because of too much of waste generation, which approx. accounts for 70% of the weight of the raw skin, there is a very large sum of money invested in the treatment of the wastage.

The current project involves the use of chrome wastage from leather industry to produce a composite material with the addition of ethylene propylene diene monomer, epoxy resin and titanium dioxide. The addition of EPDM in this composite is to increase the thermal stability of this composite when compared to the already existing compounds. The addition of EPDM alone cannot make a composite because the strength of the composite will not high enough for practical applications. This problem has been addressed by adding epoxy resin which forms the matrix for the fibers embedded in it. This improved thermal stability is evident from the TGA graphs which are discussed in detail in the results and discussion section. Whether good binding has been formed confirmed by the formation of strong double bonds, -o- linkages as given by the FTIR test. This composite formed has been further impregnated in pykrete in order to show one of its applications. The reinforced pykrete has showed improved compressive strength which is an important parameter for deciding the importance of the material as a building material.

II. LITERATURE SURVEY

A. Lorne W. Gold (2004): An investigation was done during the time of World War II to study the properties of various building materials for constructing battle combat vehicles and ships. This led to the study of reinforced ice composites as the building material for ships which was thought to be torpedo resistant.

Hence, this project gained popularity among the top brass in Britain and Canada (Winston Churchill and Lord Mountbatten). This kindled further research and development in ice mixed with wood pulp called as pykrete. Though the material had strength and toughness it was not economically feasible. Also, the lack of human labor for construction purpose resulted in the jettisoning of the project.

B. C.M. Bhuvaneswari, S.D.Kakade, V.D. Deuskar, A.B. Dange and Manoj Gupta (2008): This project deals with experimentation of EPDM (ethylene propylene diene monomer) as a thermal insulation in case bonded rockets. Here, EPDM was used as the base material blended with chlorosulfonated polyethylene to which additives in the form of fillers (fibrous and non-fibrous) were added which includes fumed silica, carbon fibers, aramid. Therefore, it is seen that the main purpose is to study the insulations for case bonded rockets.

C. S. Swarnalatha , T. Srinivasulu , M. Srimurali , G. Sekaran (2008): The presence of trivalent ions of chromium in the wastage pollutes the air and water bodies (especially ground water) due to burning the wastage and leaving untreated effluents into the river. The usual process employed to get rid of the wastage is to incinerate the wastage in minimal supply of air which is known as starved incineration. The process is carried out at a temperature of 800°c. The ash obtained from the procedure was further solidified and then tested to find out the chemical oxygen demand of chromium ions.

D. Ganjoo, R. K. , Koul, M.N. (2009): A field study and analysis was carried out in Siachen glacier (India) during summer of 2008, to record changes in the terminus of the glacier. At about 500m south of the toe of the glacier, there were proofs of glaciation in the guise of remains of valley wall striations and lateral moraine. The Siachen glacier is a geomorphological fledgling. The lateral extent of the terminus of the glacier has fluctuated within a few kilometers from the present position of the terminus. Signatures of very young fluctuations during the current geological epoch (Holocene), as recorded from elsewhere, are well preserved in close vicinity to the present-day terminus of the Siachen glacier and not 70-80 km down the valley, as reasoned on geomorphological evidences.

III. METHODOLOGY

This methodology has been prepared in compliance with the std. methodologies already existing after careful study.
Design step has been excluded because this project does not involve any design.

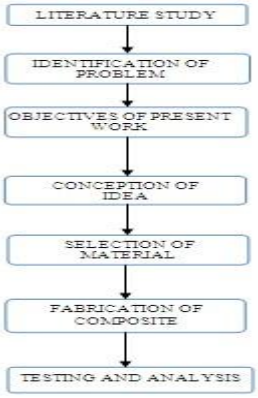

Fig. 1. Flow Chart

IV. EXPERIMENTATION WORK

Selection of Materials

Pykrete

Fig. 1. Pykrete

This material in contrast to conventional/normal ice melts very slowly and has very good mechanical properties when compared to ice. This is evident from its compressive strength which is approximately 7.3 Mpa. But these properties only exist in the solid state i.e., until the ice is below the melting point. Due to this reason buildings have been constructed in cold regions like Finland where the temperature is very low by spraying water mixed with wood pulp over inflated balloons and then spraying another layer of material till the entire structure is constructed of the required thickness. The advantage of this material is that the only primary requirement is ice which could be easily procured from the ocean and they tend to be very hard and tough resisting bullets and torpedoes during warfare. Hence, the maintenance cost of this material is very low compared to conventional materials like steel which are used to construct ships. The only disadvantage being the temperature dependency of tis properties i.e., they tend to exhibit the properties only under their melting point.

EPDM (Ethelene Propylene Diene Monomer)

EPDM has been researched for its capability as an insulator in rockets to protect the material of the rocket from the intense heat produced during the combustion process. This property of the material due its low thermal conductivity has been utilized in this project to give the composite the ability to prevent heat from passing through the material. The glass transition temperature might be decreased due to the presence of rubber which softens easily at low temperatures. But it has been shown that the glass transition temperature is well beyond the room temperature making a hard, brittle material at the room temperature range This material has been investigated by DRDO already for the application of EPDM based insulation system for case bonded rockets. This gives us some confidence in using this material as insulation material in our composite. But this is a component that can decrease the glass transition temperature because it has a low T_g value. This could elicit suspicion that the material may become a rubbery, soft substance at temperatures slightly above room temperature. But this has been ruled out by DSC test which shows a T_g value of around 56˚c.

Fig. 3. EPDM (Ethylene Propylene Diene Monomer)

Buffing Dust

In order to improve the thermal properties (thermal conductivity) of pykrete EPDM rubber must be impregnated in the above said material. But there is an inevitable decrease in the mechanical properties of the pykrete composite. So, in order to improve the thermal properties without any significant effect on the mechanical properties buffing dust-EPDM composite material is used because already existing research papers have shown that buffing dust, as the filler material in the composite ,improves the mechanical properties which needs to be further investigated and analyzed for this particular composite**.**

Epoxy Resin

In order to bind the EPDM rubber and buffing dust, Epoxy resin is used as the matrix which holds them both together in the composite material. In addition to all these materials Titanium dioxide Nano powder with particle size range of 3540nm has been added to achieve improved properties.

Titanium dioxide

Titanium Dioxide has been incorporated in one of the compositions to improve the UV (ultraviolet radiation) resistance property of the composite. Titanium dioxide is a

Fig. 4. *Buffing Dust*

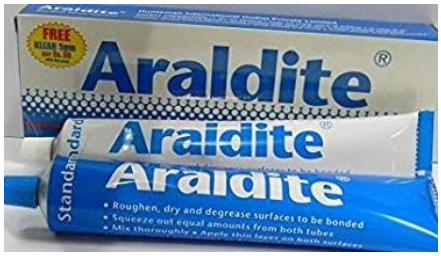

Fig. 5. Epoxy Resin and Hardener

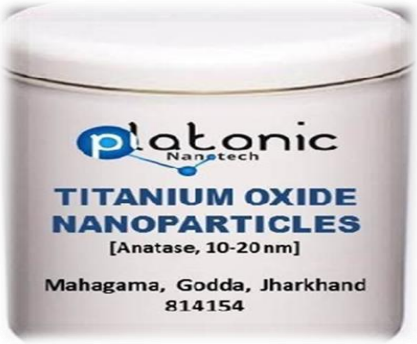

Fig. 6. Titanium Dioxide (Anatase)

semiconductor material with the gap between its valence and conductivity bands much more when compared to many other metals. This protection against ultraviolet rays could extend the application of this composite for helmets for two wheelers which could protect the skin from harmful UV A and UV B radiation In this particular testing and analysis one of the compositions is incorporated with titanium dioxide to reap the benefits of it. This is evident from the lighter coloration of the composite in comparison to other compositions.

Fabrication

Polymer matrix was prepared by mixing epoxy polymer (araldite) and hardener. The resin and hardener were low viscous to achieve easy workability. Leather waste has been obtained from the tannery section of a leather industry in chrompet. To remove the moisture from the leather waste, it has been heated to 100 degree Celsius for 3 hours until the moisture dropped to 20% on wet basis and remained constant.

Preparation

The resin and hardener were taken in different proportion and they are mixed continuously in a magnetic stirrer. On mixing it polymerizes to give a high-density fluid. After homogeneous mixing for ten minutes the leather waste was added and again mixed for ten minutes. Leather waste reacts along with the ongoing process to give a composite product in semi-solid form. In case of addition of filler material, it was mixed well with the leather waste prior to mixing with the resin mixture.

Curing

The mixture was transferred on a plastic sheet and compressed into a sheet of thickness less than 4mm using a pressing machine. After which the mixture was kept in a oven for 3 hours and heated for about 70 degree Celsius. After complete curing for 3 hours the material is stored for. Around one week to only the material to completely cure under ambient conditions. if the material is not given this time period to cure and is immediately tested to find out the results then the results may give unexpected values ending in erroneous results. In order to avoid such avoidable errors, the material has been stored in room temperature for around one week time post which the material was tested.nCuring can be carried at various temperatures, but the time

TABLE 1. Various Compositions of composite

Sample	Buffing Dust	EPDM	TiO2	Temp	Epoxy resin
S1	1	3	-	100˚c	10
S2	1	1	0.05	HT	10
S3	0.5	1	-	HT	10

required for curing will vary accordingly. For instance, at 100°c the time required is 1 hour whereas at 70°c the time required is 3 hours. Depending on the rate of curing the reactions also differ and hence, the properties.

V. TESTING

A. *Thermo Gravimetric Analysis*

The thermal stability was evaluated for Leather particulate (buffing dust) – EPDM systems prepared. To establish its thermal properties thermo gravimetric analysis and differential scanning calorimetry has been performed. The temperature decomposition curve has been obtained to understand how the material behaves under thermal changes when the polymer and the leather waste have formed a composite. The analysis was carried out according to ASTM E1131:2008 (Ra 2014) standard. This procedure is carried out to ascertain the highly volatile matter, medium volatile matter, combustible material and ash content.

B. *Differential* Scanning *Calorimetry*

Differential scanning calorimetry [DSC] was carried out under Nitrogen at atmosphere at heating rate of 10°C/min to analyze resin curing.

The thermal behavior studies of the polymer blends could be one of the most effective methods for determining the miscibility of blends. The extent of cured network structures formed could also be directly read from the glass transition temperatures of the cured resins. When two polymer blends are are miscibilised at the molecular level, a single and composition dependent Tg is observed between the Tg's of the blend components. In case of poor miscibility between the matrix and the fiber, there exist two or more recrystallization temperatures, the range of which indicates the degree of miscibility. This procedure was carried out according to ASTM D7426: 2008 (Ra 2013) standard. this method when carried out under various conditions gives various results.

C. *Fourier Transform Infrared Spectroscopy*

In order to understand the chemical composition of the composites Fourier transform infrared spectroscopy has been performed. From this it is understood if there has been any interaction between the leather particulate, EPDM and epoxy matrix. The bonds formed between the various elements present are considered as springs which when subjected to a force oscillates back and forth. Absorption of certain wavelength (wavenumber) of IR radiation indicates the presence of a particular bond. Hence, this can be used as fingerprint for the identification of the synthesized compounds by comparing with IR spectroscopy results of existing known compounds from the library. For instance, a peak in the range of 3000 cm–1 indicates the presence of C-H sp^3 hybridized bond. Similarly, all other bonds can be identified by referring to the existing tables. But since the shape and intensity of the absorption peaks must also be considered, it becomes a hectic task to ascertain the molecular structure of the compound. Therefore, software has been developed to identify the correct bond by using various algorithms.

D. *Density*

Density is measured by obtaining the volume of the sample, which is length x width x thickness, and converting this into cubic centimeters. The weight of the sample in air, measured in grams, divided by the volume, in cm3 , gives a density presented in grams per cc, which is cubic centimeter. This procedure was carried out according to the ASTM D792 standard.

VI. RESULT AND DISSUSION

A. *Thermo gravimetric Analysis*

The variation in the weight ratio of Leather particulate - polymer -EPDM did not affect the thermal stability of the composites. The temperature at which 90% weight loss occurs around 340^0 C. The aliphatic groups in the resin might have reacted with the collagen network in the fibers which contributed to higher decomposition temperatures. Also the metal ions inherently present in the leather waste could have supported the fact of higher decomposition temperature.

The modified composites with Filler (Titanium Dioxide) show an increased TGA value of 350^0 C. The marginal rise was due to the very small addition of Filler.

The material remaining behind after exposing the sample to nitrogen, which the TGA shows comprises 10% of the mass of the polymer. The sample has been tested according to the ASTM standard in order to comply with the requirements of any industry in which the material might be used in the future. Hence, the tests were carries out in NABL lab in Vanagaram in accordance with requirements of the standard. A standard temperature of 25°c was maintained. The TGA graph has proven to be very useful in studying the thermal properties of the material. The material as discussed above has shown very good thermal properties in addition to the chemical bonding between its constituents, high glass transition temperature, low density and very good hardness. As usual this shows that not only the material can be used as a building material but it can also be used as a refractory material in furnaces and kilns due to its high decomposition temperature in the range 0f 350°c . This can further be improved by changing the ratio of the various constituents in the mixture and finally finding out the optimum composition at which the material will show much pronounced properties but needs more investment as well as lot of intensive research.

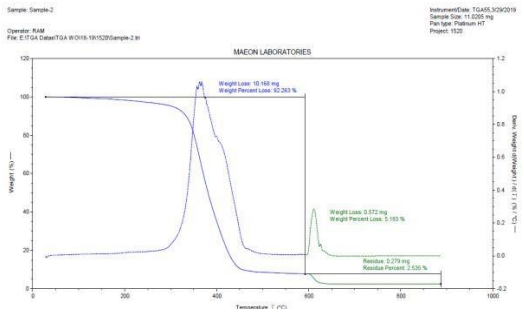

Fig.7. TGA GRAPH (B)

The graphs for the Thermo Gravimetric Analysis (TGA) for the already tried composites made of leather waste show a decomposition temperature of just 230 °c as shown by the graph adjacent to the graphs for the newly fabricated composite.. But as far as our composite is concerned the decomposition temperature has invariably been increased to 350 °c as is evident from the graph shown below where a straight line indicates the inflection.

In the graph below for already existing composites made of leather waste at various proportions of the leather waste in the epoxy polymer has been evaluated. The decomposition temperature is indicated by means of the straight line at the point of inflection. It is evident from the graph that the temperature of decomposition for already existing compositions is

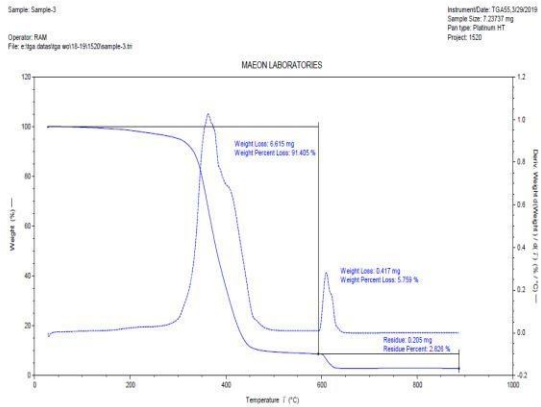

Fig. 8. TGA Graph (c)

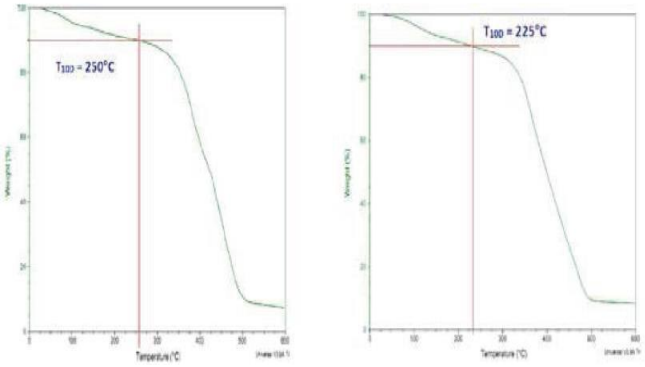

Fig. 9. TGA Graph (Literature)

much lower than the newly made composite. The difference is 100°c between the two compositions i.e., old one and the new one.

B. *Fourier Transform Infrared Spectroscopy*

There are many polymers with different structures, different polymerization degrees etc. IR spectroscopy can be used to characterize the nature of the polymer. The above mentioned FTIR graph was obtained in the earlier investigations of the sample without the addition of EPDM filler . the following three graphs are of the present work which serve as a fingerprint in the identification of the various bonds formed in the structure.

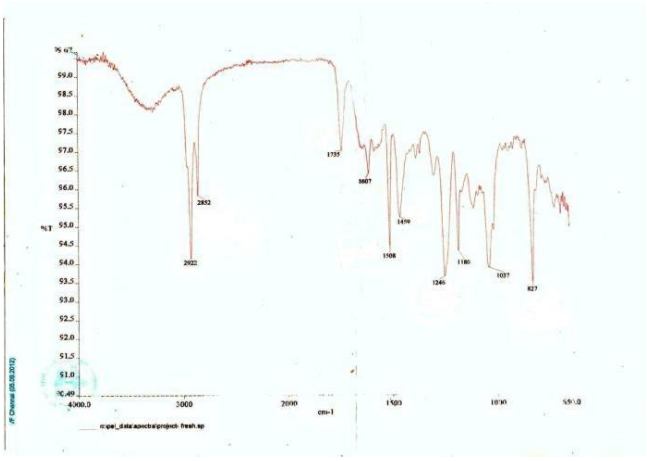

Fig. 10. FTIR graph (literature)

The spectra of leather particulate -polymers are given. The presence of epoxy group was confirmed by the presence of bands around 1249 cm^{-1} and 830cm^{-1}, which accounts for the symmetrical and asymmetrical stretch respectively. The presence of C-H sp^3 hybridized bond was confirmed by the absorption peaks occurred at 2926 cm^{-1}. the presence of several peaks of medium intensity in the range of 1250-1050 cm^{-1} indicates the presence of C-O-H stretch. The presence of signals at 1649 and 1612 cm^{-1} indicates the presence of C=C. intensity of the signal in this case is low indicating a weak dipole moment. Weak dipole moment is attributed to the donation of electrons by the alkyl group and asymmetric structure. If it would have been a symmetric structure around the double bond , then the electrons donating effect of the –R groups on both the sides would be the same and there would be no dipole moment indicated by an absence of signal in this 1600 cm^{-1} wavenumber region. The FTIR spectral analysis showed that the Leather particulate - Polymer formulations have been cross linked effectively. The above-mentioned peaks and their corresponding groups have been tabulated in the following table for sample 1 for easier understanding of the scenario. In comparison to the earlier work, the present work has performed well which is evident from the intensity of the peaks in the graph. As far as the second and third fraps are concerned they almost show the same peaks in the same band ranged but with increased intensity resulting in the.

TABLE 2. FTIR Analysis

FTIR PEAKS (cm^{-1})	ASSIGNMENT OF GROUPS
2926	C-H STRETCHING (sp^3 hybridized)
1249	EPOXY GROUP (O-O)
1182.36	C-N (leather fiber)
1649 ,1612	C=C (asymmetric)
1103, 1182,1033.8	STRETCHING OF C-O-C
<1000	METAL IONS

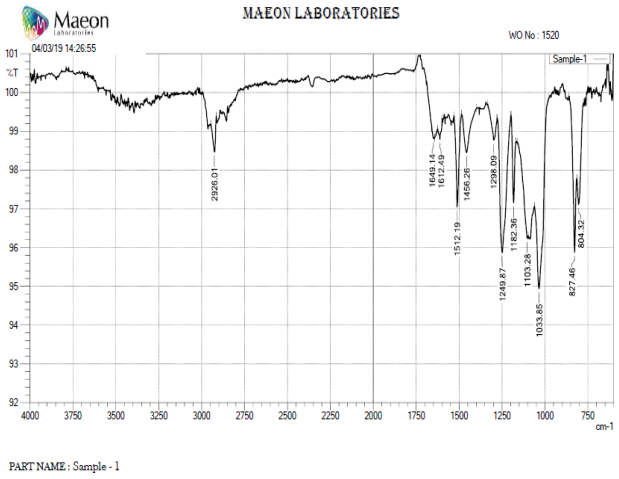

Fig. 11. FTIR Graph (A)

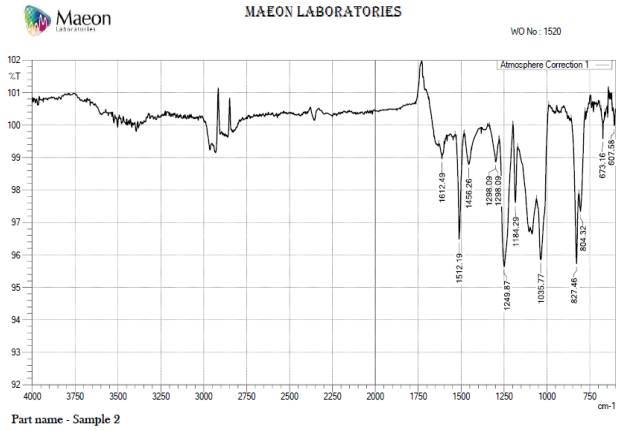

Fig. 12. FTIR Graph (B)

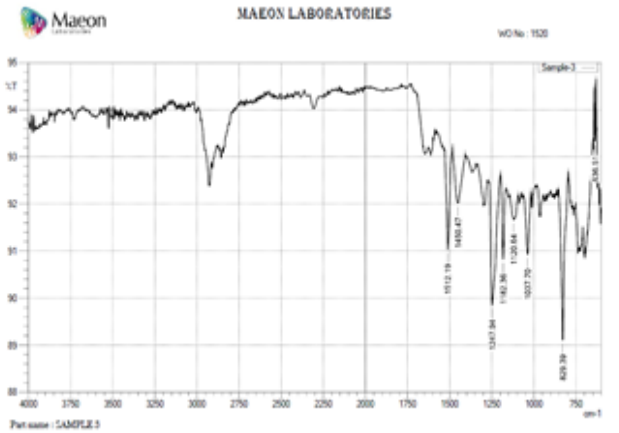

Fig. 13. FTIR Graph (C)

The chromium ion which is present in abundance in buffing dust is responsible for the formation of such peaks in the region lesser than $1000cm^{-1}$ in this case. Hence, this behaves as proof for the presence of chromium which is otherwise toxic and causes the pollution of rivers and other bodies in which these wastes are dumped. As far as the second and third graphs are concerned, they also show almost the same absorption peaks and hence proves that the bond formed between various constituents in the blend is almost the same in all of the compositions irrespective of any change in the proportion of various constituents. Therefore, this material proves to be a good composite material which can further be analyzed for its mechanical properties FTIR test is basically done to prove the existence various bonds between the constituents when a new/novel material is fabricated. The test accordingly with the aim of proving the existence of various bonds has turned out to be a great success.

C. *Differential Scanning Calorimetry*

The glass transition temperature obtained from differential scanning calorimetry is one of the important tools to understand whether there is good miscibility of the components. As discussed in the testing chapter the graph shows that there is only one glass transition temperature which indicates the homogenous mixing of the components with each other. The glass transition temperature is marked by downward inflection as shown in the illustrative graph for comparing purposes. The downward inflection is due to the endothermic reaction. It is followed by the peak which gives the crystallization temperature where the material which is either in amorphous state or semi crystalline state is transformed into crystalline state. In amorphous state the molecular chain is randomly oriented which undergoes orientation in a direction which in turn releases heat and therefore happens to be an exothermic reaction. Glass transition temperature is the temperature at which the polymer changes from hard, glassy state to rubbery state. Since, the material being fabricated has glass transition temperature well above the room temperature the material will remain in its hard state. The glass transition temperature of the composites is 57°c, 54°c and 59°c respectively.

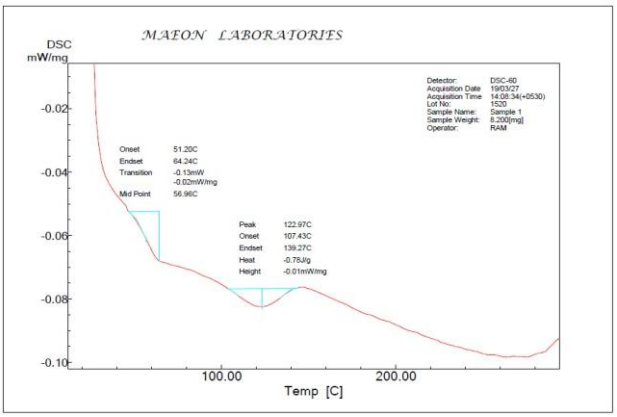

Fig. 14. DSC Graph (A)

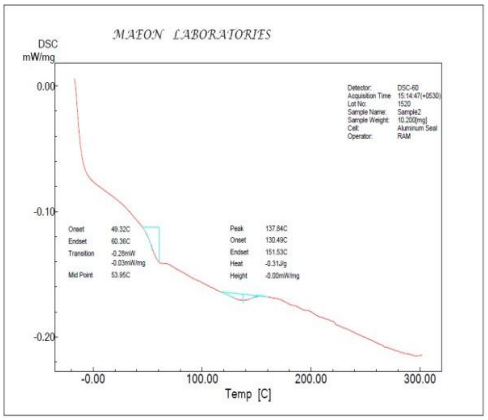

Fig.15. DSC Graph (B)

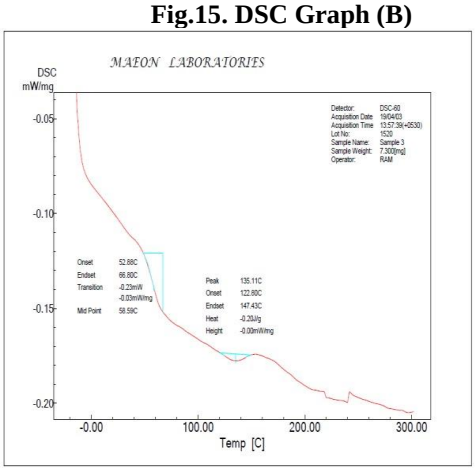

Fig. 16. DSC Graph (C)

D. *Density*

Density of the composites decreases with the decreasing trend of the leather dust content. This could be attributed due to the fact that density of the leather dust is much less in comparison with the polymer matrix. This fact leads to greater end of these leather composites in case of light weight applications. On reducing the leather waste content the value of the density further drops. On adding Filler density of the material has dropped significantly with may be due to the fact it has blocked the sites available for polymerizing with eventually lead to lowering of density.

E. *Compressive strength*

The compressive strength of pykrete material after embedding it with buffing dust – EPDM composite was found out to be 8.3 Mpa , 8.2 Mpa and 8.6 Mpa which is well above the compressive strength of normal pykrete.

VIII. FUTURE SCOPE

The material was fabricated with construction on glaciers and other cold regions as the major targets. But this material could be used in a wide variety of applications making it a versatile composite material not only applicable for building constructions but could also be used in bullet proof vests, reinforced ice roads in cold regions but requires further research and analysis. This material could also be used in other applications such as building restaurants and other similar recreational or amusement buildings in the cold regions which would provide the customers with a warm environment on the icy cold regions. There are many advantages of usin this material in the military area not for construction of buildings but also for the construction of lamps which hitherto does not exist.

IX. CONCLUSION

Leather particulate – polymer composite modified with fillers (titanium dioxide) have showed good performance with good thermal properties.

This property has been supported by the strong mechanical phase interaction brought by the chemical bonding with leather interface was confirmed by FTIR analysis and Thermo Gravimetric Analysis.

Thermo Gravimetric Analysis revealed the higher decomposition temperature for the composites with approximately equal but higher than the already existing leather – polymer composite.

From all the above-mentioned features the composite can find its application in the Siachen glacier. The hardness of the pykrete material enhanced with buffing dust – EPDM composite has shown better hardness and hence, it could be used as a building material. The light weight these composites show that they provide strength with reduction in the weight of the 'Pykrete' composite.

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AUTHORS PROFILE

Manikandan G M.E., Computer Integrated Manufacturing

Srinath R B. Tech, Mechanical Engineering

Akshay Naramshettiwar B. Tech, Mechanical Engineering