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RESEARCH ARTICLE

STUDY ON SEDIMENTATION STABILITY OF MAHUA AND SIMAROUBA OIL BASED MAGNETORHEOLOGICAL FLUIDS

Ram Rohit Vannarth[1*], K. Mallikharjuna Babu[2], P. Martin Jebraj[3]

[1]Department of Mechanical engineering, BMS College of Engineering, Bengaluru, India.
[2]Department of Industrial Engineering, BMS College of Engineering, Bangalore, India.
[3]Department of Material Science and Engineering, Adama Science and Technology University, Adama, Ethiopia.
**Corresponding author email: ramrohit.mech@bmsce.ac.in*

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ABSTRACT

Magnetorheological fluids (MRFs) are peculiar of smart fluids whose rheological properties can be efficiently controlled by varying applied magnetic field. Owing to this peculiar property, it can be utilized in industrial applications in semi-active devices such as dampers, actuators, etc. In this work, an effort has been attempted to produce MRFs using natural oils as carrier fluids and tested for potential replacement of conventional carrier fluids such as silicone oil. The MR fluids are prepared using silicone oil and two natural oil namely Mahua oil and Simarouba oil. Carbonyl iron (CI) and Electrolytic iron (EI) are used as suspended particles with lithium grease as an additive. The MRF using two natural based oil are known as Green magneto rheological fluids (GMRF). The characterization of suspended particles and carrier liquids was performed, followed by tests on sedimentation stability of prepared samples. To compare the sedimentation stability, the graphs of sedimentation ratio versus time are plotted for all MR fluids samples. It is found that the sedimentation stability of natural oils based MRFs are relatively better than silicone oil based MRFs for the same concentration of magnetic particles. There is potential of these natural oils based MRFs to replace the conventional MRFs However, other rheological properties like yield strength, flow characteristics etc. needs further investigation.

KEYWORDS

Magnetorheological fluids, Green Magnetorheological fluid, Natural oils, Mahua oil, Simarouba oil, Sedimentation stability.

1. INTRODUCTION

Magnetorheological fluid (MRF) is a smart material whose behaviours such as shear viscosity and yield strength can be changed significantly due to its responsiveness to an applied magnetic field [1]. It is made up of magnetic polarizable particles and non-reactive and non-magnetic carrier fluid along with coating [2]. MRFs behave almost like Newtonian fluids whose off-state viscosity are determined by the carrier fluid and the number of polarizable particles [3,4]. When the external magnetic field is applied on the MR fluid, the polarizable particles aligned along the magnetic field forming chain like structures thereby behaving like a quasi-solid instantaneously [5-8]. Magnetorheological fluids should not be confused with similar smart materials such as ferrofluids which have Nano sized particles making it nearly 1000 times smaller than the particles used in MRF. The ferrofluids have similar composition, but due to Nano sized particles in dispersion, they do not show profound viscoplastic effects like MR fluids [3,8]. The ER fluids have similar working principle except for electrically controlled rheological properties instead of the magnetic field [9]. This exclusive property of MRF is utilized in many commercial applications of semi active devices such as dampers, clutches, actuators and other applications such as medical applications, glass polishing and many more [10-19]. The rheological properties of MRFs depend on the type of suspended particles used and their concentration and the applied magnetic field [20]. Different kind of magnetic particles has different shapes and structures. These morphological properties of the particles also affect the yield strength developed in the MRFs thereby limiting the controllability and usability of the MR fluids [21-23]. Controllability of MRFs depends on the response of the suspended particles to the external/applied magnetic field. To achieve high sedimentation stability, the size of magnetic particles must be very small, generally in the range of 1 μm to 10 μm.

In general, the commercially available MR fluids have synthetic oil, mineral oil or silicone oil as the base fluid, and carbonyl iron (CI) or electrolytic iron (EI) as magnetic particles. Carbonyl iron (CI) particles are most commonly used as magnetic particles because of their high saturation magnetization (M_s) [24,25]. Synthetic oils have very desirable properties such as high flash point, high shear strength, high viscosity index and low friction. Besides, they do not stiffen at high temperature. But the cost of synthesizing this oil is quite high [26]. On the other hand, mineral oils have low flash and fire points thereby limiting it to the use of low temperature applications [27]. and silicone oil has low surface tension and wets the surface quickly which leads to accumulation of dirt [28, 29]. In typical MR fluids, surface coatings/additives are used to prevent conglomeration of the magnetic particles by acting as surfactants.

A magnetorheological fluid is more prone to sedimentation than the ferrofluids due to the density mismatch of suspended particles and carrier fluid and also due to the Brownian motion fiasco to keep the particles suspended. Agglomeration and sedimentation has been a challenge and unfavourable in potential application of MRFs and needs to be addressed. Sedimentation rate depends on various factors such as weight fraction of particles, the density of carrier fluid and particles, adhesiveness and size and shape of particles [30, 31]. Hence to improve the sedimentation stability, use of thixotropic agents such as silica, carbon fibers are prescribed [32]. Use of surfactants like oleic acids, which prevent agglomeration of particles, or using Nano-sized particles to increase

dispersibility is found to be advantageous [33,34]. In this current research work an attempt to address this problem has been made.

The present work is an effort to investigate the sedimentation stability of natural oil-based MR fluid and compared with the conventional silicone oil-based MR fluids. The various researches have been made to improve the characteristics of MRF by using alternate base fluids with different suspended particles and additives. In this work, MRFs are synthesized using Mahua and Simarouba oils which are extracted from seeds of Mahua tree (*Madhuca longifolia*) and Simarouba tree (*Simarouba amara*) respectively. CI and EI particles are used as the particles. Lithium grease is used as additives. Similarly, samples of MRFs using silicone oil are also prepared. More specifically, the handouts of this work are summed up as follows: (1) finding the factors affecting the sedimentation stability of Mahua and Simarouba oils based MRFs, (2) comparison between the sedimentation stability of the natural oils based MRFs and silicone oil based MRF. In order to find out all these objectives, the various properties of the oils and the suspended are examined through various tests.

2. CHARACTERIZATION OF OIL

Cold pressing methods are used to extract the oil from mahua and simarouba kernels. By converting the seeds into a fine paste, it was stirred using rotating screw and the oil is collected. The entire process was carried out in isothermal conditions. After extraction, the densities of the oil were checked using a hydrometer. The densities of mahua oil and simarouba oil were found to be 956 kg m^{-3} and 966 kg m^{-3} respectively while the density of silicone oil was found to be 959 kg m^{-3}. The high density of the simarouba oil is attributed to the triglyceride chain in the molecular composition [35].

In order to check the volatility of the oil samples, the flash and fire points of the oils were checked. If the flash and fire points of the oils are low, they cannot be used for high temperature applications. The flash and the fire points were determined using the Pensky-Martens Closed Cup Apparatus (IS:1448 (Part-66)). This apparatus is used because of its ability to detect contaminants and higher precision. The sample is initiated into the cup and to the top of the cup, a close-fitting lid is provided. The cup containing the test sample is then heated and stirred. After some time (approximately 2 minutes), the aperture is opened in the lid so that the ignition source can be dipped into the vapors to test the flash point. After every 5 °C change in temperature, the same procedure of opening the aperture and dipping the ignition source is repeated so that the lowest temperature at which there is momentary fire or flash can be noted down. After finding the flash point, the test sample is continued heating until the oil catches continuous fire of about 5 second. This temperature at which oil begins to burn is recorded. This gives the fire point of the oil.

Shear viscosity of MR fluids depends on the carrier fluid and the number of particles used [25]. Hence the viscosities of the oils are needed to be determined. The kinematic viscosities of the oil were measured in the temperature range of 40 °C to 100 °C. In this work, capillary U-tube viscometer is used at 40 °C. The sample is introduced into the capillary tube in the lower bulb up to a level which is marked. The sample is then pulled up to the topmost bulb which is immersed in a thermostat bath to maintain a constant temperature. After the specific temperature is reached, the suction of the sample is stopped and allowed to flow down through the capillary tube. The time taken by the volume of the fluid to pass through the intermediate bulb in the capillary tube is noted down. Kinematic viscosity is found out by multiplying viscometer constant with the time taken by the volume of the fluid to pass through the intermediate bulb.

To determine the calorific value of the oil, bomb calorimeter is used. First, the energy equivalent of the calorimeter is determined empirically by burning a standard substance of known heat capacity under controlled conditions. Then the test samples were weighed and burned. The rise in temperature of the test sample is measured and the value is noted down. By multiplying the energy equivalent of the calorimeter with the resultant temperature rise, the amount of heat obtained is determined. Hence the calorific value of each sample is determined by dividing the amount of heat obtained by the weight of the test sample.

Table 1 gives details about the properties of oils used as carrier fluids in this work.

Table 1: Properties of the oils

Properties	Units	Silicone oil	Mahua oil	Simarouba oil
Density at 30 °C	kg m^{-3}	959	956	966
Kinematic Viscosity at 40 °C	Mm^2 s^{-1}	54	52	56
Flash Point	°C	110	142	145
Calorific Value	MJ kg^{-1}	23.24	37.00	36.24

3. CHARACTERIZATION OF PARTICLES

3.1 Scanning Electron Microscopy (SEM) Analysis

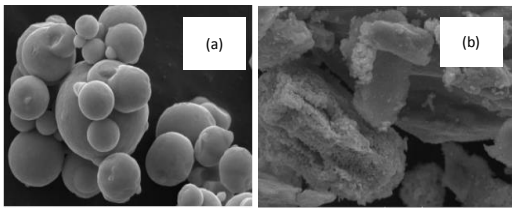

Figure 1: (a) SEM image of CI powder at 15000 X magnification; (b) SEM images of EI powder at 15000X magnification

The structure of the magnetic particles used in the MRFs also affects the strength of the fluid. To examine the morphological structure of the particle at a high resolution up to 50 Å, SEM analysis is carried out. In this, specimens of the magnetic particles were exposed to high accelerated electron beam (~ 30 kV) from an electron gun, which scans the surface of the specimen rapidly. This results in the release of primary electrons, secondary electrons, Auger electrons with X rays, from the surface of the specimen, which act as source for imaging. These electrons are accumulated and detected by a detector which in turn produces electronic signals. The image was captured using a digital camera and was processed by a computer. TESCAN, Scanning electron microscopy(SEM) was used to carry out the test.

3.2 EDAX Analysis

Energy Dispersive X-Ray Analysis (EDAX) was used for the chemical characterization of iron particles. Element has a unique atomic structure and therefore shows a particular set of peaks on its X-ray emission spectrum. An atom of an element has different energy levels for its electrons. When an electron is excited, it shifts from its lower energy level (ground state) to a higher energy state and an electron from the higher energy shell jumps to lower energy shell thereby creating an energy difference which is emitted in the form of X-rays. EDAX software analyses the emitted rays and gives the relative composition of the sample in graphical form. K-Ratio denotes weight fraction of the material, is defined as the ratio of unknown intensity to reference intensity.

Table 2: Chemical composition of CI powder from EDAX

Element	Weight Percentage	Atomic Percentage	Net Intensity	Error Percentage	K Ratio
Mn	3.54	3.60	3.35	20.96	0.0389
Fe	96.46	96.40	323.99	2.49	0.9883

Table 3: Chemical composition of EI powder from EDAX

Element	Weight Percentage	Atomic Percentage	Net Intensity	Error Percentage	K Ratio
C	6.95	25.73	5.77	18.89	0.0190
Si	0.04	0.07	0.33	99.99	0.0002
P	0.09	0.12	0.75	72.26	0.0005
S	0.14	0.20	1.57	68.30	0.001
Cr	0.53	0.45	6.51	48.14	0.0080
Mn	0.81	0.65	6.45	49.77	0.0089
Fe	90.57	72.16	584.58	2.08	0.9078
Cu	0.88	0.61	3.39	61.19	0.0076

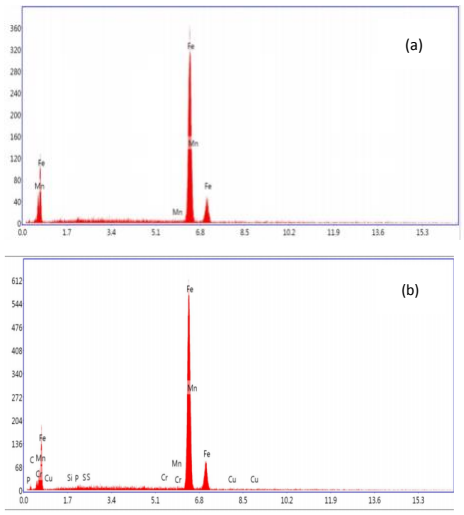

Figure 2: (a) Graph obtained for CI powder in EDAX; (b) Graph obtained for EI powder in EDAX

3.3 Vibration Sample Magnetometry

To examine the magnetic behavior of the iron particles, vibration sample magnetometer (shown in figure 3) is used. The sample iron particle is subjected to mechanical vibrations under a constant magnetic field. The VSM detects a voltage harmonious to the magnetic moment of the iron particles. By doing so, magnetic moments of the particles for the corresponding magnetic field were determined and the hysteresis curves (M–H) of the particles were plotted as shown in figure 4. In our research work, the magnetic behaviour of the particles is measured at 298 K and 400 K.

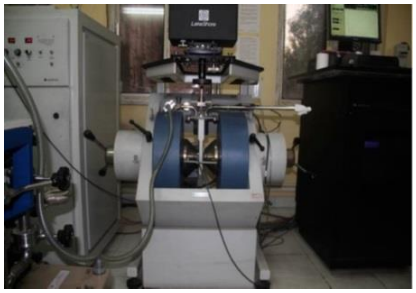

Figure 3: Vibration Sample Magnetometer

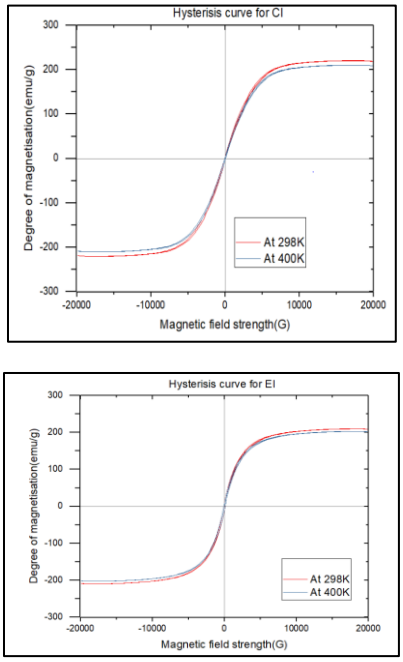

Figure 4: (a) M-H Curve for CI particle; (b) M-H curve for EI particle

3.4 Sedimentation

In our work, carbonyl iron (density: 7.86 × 10^3 kg m^{-3}, CN grade, BASF grade) and electrolytic iron (density: 7 × 10^3 kg m^{-3}, CN grade) were used as base particles. They are ferromagnetic in nature. Three different carrier fluids were used: Silicone oil, Mahua oil and Simarouba oil. To find how the stability of MRF and GMRF varies at different volume fractions, a series of MRF and GMRF samples were prepared. Lithium grease was used as an additive in all samples. In order to check how the stability of MRFs and GMRFs changes due to the change of volume fraction of base particles, three samples were prepared with the same carrier fluids (say silicone oil) and same base particles (CI particles) but different volume fractions (20 wt.%, 30 wt.% and 40 wt.% base particles and 10 wt.% additives in all cases).

In order to mix the different components properly, the samples were stirred using a mechanical stirrer for about 30 minutes. In addition, to test how the stability of the fluid changes due to the change of base particles, three more samples were prepared using electrolytic particles that is, replacing the CI particles in all the previous cases with electrolytic particles. Hence, six samples of MRFs were prepared by combination of two base particles (CI and EI particles) and one carrier fluid (silicone oil). Similarly, twelve samples of GMRFs were prepared for mahua oil and simarouba oil as carrier fluid. The prepared MRF samples were poured into the glass tubes. The tubes were placed without any disturbance for several days and observed the change in the contents of the tubes. The stability of the MRF and GMRF was measured in terms of sedimentation ratio which is defined the as ratio of height of supernatant liquid column above the mudline to the height of the fluid in the tube, and time required for the sedimentation is measured in hours. The sedimentation is due to pure gravitational force (no centrifugal action or imposed magnetic field for faster sedimentation). Sedimentation ratio is defined as,

$$\text{Sedimentation ratio (S.R.)} = \frac{\text{Height of liquid column above mudline (supernatant liquid)}}{\text{Total height of liquid column}} \quad (1)$$

Table 4: Nomenclature of prepared GMRF and MRF samples

Sl.no	Samples	Carrier fluid	Particles
1	MRF SC 20	Silicone oil (70 wt.%)	Carbonyl iron (20 wt.%)
2	MRF SE 20	Silicone oil (70 wt.%)	Electrolytic iron (20 wt.%)
3	GMRF MC 20	Mahua oil (70 wt.%)	Carbonyl iron (20 wt.%)
4	GMRF ME 20	Mahua oil (70 wt.%)	Electrolytic iron (20 wt.%)
5	GMRF SC 20	Simarouba oil (70 wt.%)	Carbonyl iron (20 wt.%)
6	GMRF SE 20	Simarouba oil (70 wt.%)	Electrolytic iron (20 wt.%)

Table 4 gives details about the nomenclature of samples and respective interpretations. All samples are constituted of 10 wt.% lithium grease as an additive. The sedimentary velocity of the samples was calculated using stokes equation and tabulated in table 5 below. The sedimentary velocity of GMRF and MRF samples were calculated by Stokes equation 4.8 given below.

Table 5: Sedimentation velocity of various GMRFs and MRFs.

Sl. No.	Sample	Diameter of particle (m)	Viscosity of carrier fluid (Pa. s)	V_{sed} (m s^{-1})
1	GMRF MC 20	6 ×10^{-6}	0.0498	2.716 × 10^{-6}
2	GMRF ME 20	10 ×10^{-6}	0.0498	6.715 × 10^{-6}
3	GMRF SC 20	6 ×10^{-6}	0.0543	2.485 × 10^{-6}
4	GMRF SE 20	10 ×10^{-6}	0.0543	6.141 × 10^{-6}
5	MRF SC 20	6 ×10^{-6}	0.0519	2.605 × 10^{-6}
6	MRF SE 20	10 ×10^{-6}	0.0519	6.439 × 10^{-6}

$$\text{Sedimentary velocity } (V_{sed}) = \frac{(\text{Density of magnetic particles} - \text{Density of carrier fluid}) \times g \times d^2}{18 \times \eta} \quad (2)$$

where V_{sed} is the sedimentary velocity in m s^{-1}
ρ_p and ρ_c are the density of magnetic particle and density of carrier fluid in Kg m^{-3} respectively
g is acceleration due to gravity equal to 9.91 m s^{-2}
d is the diameter of the magnetic particle in meter
η is the dynamic viscosity of carrier fluid in Pas

4. RESULTS AND DISCUSSION

Traditional MRF uses silicone oil as carrier fluid. They have a broad operating temperature range i.e. High flash point 110 °C which is suitable for high temperature application. Flash point of mahua and simaroubha oil were 142 °C and 145 °C which is much higher than silicone oil which is highly desirable and hence makes mahua oil and simaroubha oil an alternative and right contender for high temperature MRF application. The kinematic viscosity @ 40°C and density @15°C of Mahua and Simarouba oil is 52 cSt, 56 cSt and 956 Kg m^{-3}, 968.04 Kg m^{-3} respectively which is lower than the kinematic viscosity @ 40°C and density @15°C of silicone oil evincing a paramount MR effect and high speed of response.

The structure of CI particle was found to be spherical with smooth surface. Due to the spherical shape, the bond between the particles is more in case of CI particles thereby increasing the yield strength developed in MR fluids [36]. The average size of the CI particles was found to be 4 µm (shown in figure 1 (a)). EI particles are irregular flaky shaped with diverse aspect ratio and the average size of the particles were are 10 µm (shown in figure 1 (b)). The X-ray diffraction patterns of carbonyl iron particles (CI) and electrolytic iron particles (EI) are disclosed in Figure 2 (a) and Figure 2 (b) respectively. For both CI and EI particles XRD results affirm the presence of unblemished phase α-Fe (body centered cube).

The saturation magnetization (M_S) of the CI and the EI particles at 298 K were found to be 220 emu g^{-1} and 210 emu g^{-1} respectively. There is a slight drop in the magnetization saturation at the elevated temperature. At 400 K, the Magnetic saturation values were found to be 210 emu g^{-1} and 204 emu g^{-1} for CI and EI particles respectively. The reason for the drop of the magnetic saturation at elevated temperature is due to the thermal agitation of the particles as there is an increase in thermal energy, hence decreasing the polarizability.

Generally, the suspension undergoing sedimentation consists of three unique parts: a supernatant zone which is the topmost part, the concentrated suspension which is very thin and just below the mudline and the highly packed sediment part which is the bottom most part. The previously defined sedimentation ratio cannot give entire information about sedimentation stability as it is only the relative volume of highly packed sediment in addition to concentrated suspension, therefore, cannot give information about the position of the colloidal particles [30], but it is used because it can be applied for different types of suspension system [30]. Sedimentation stability is one of the main characteristics of MR fluids. It gives the performance parameter of the fluid.

Various factors like an external magnetic field, shear rate, temperature, yield strength, etc. also have influenced the overall stability of the MRF samples. However, in our work, the MRF samples are tested only for their sedimentation stability and their results are compared. The height of the supernatant is measured (centimetre) through virtual observation of the mud line travel. The mud line travel was observed at regular interval of time and calculated the sedimentation ratio with respect to time using equation (1). The graphs of sedimentation ratio vs time are plotted for each sample, shown in figures 5 and 6.

4.1 Effect of base oil on the sedimentation stability

When the MRFs are left and allowed to settle only under the action of gravity, the particles in MRFs using Mahua oil as the carrier fluid settles quickly as compared to those in the MRF samples using simaroubha oil as the carrier fluid and silicone based MRF. From the graphs of figure 5, it is found that compared to mahua oil and silicone based MRFs, the sedimentation of the particle with respect to time is less in case of simarouba based MRFs. This is because the density of simarouba oil (966 Kg m^{-3}) is greater than that of the silicone oil (959 Kg m^{-3}) and mahua oil (956 Kg m^{-3}). As a result, the size difference between the suspended particles and the carrier fluid becomes lesser and hence it takes longer time for particles to settle down in simarouba oil. From the figure 5 and 6, it is seen that for the same weight percentage of suspended particles, it takes longer time to settle for the particles in GMRF using natural oils than those in the MRFs using silicone oil and mahua based GMRF. Hence the settling rate of simaroubha oil based GMRFs are less thereby less sedimentation ratio.

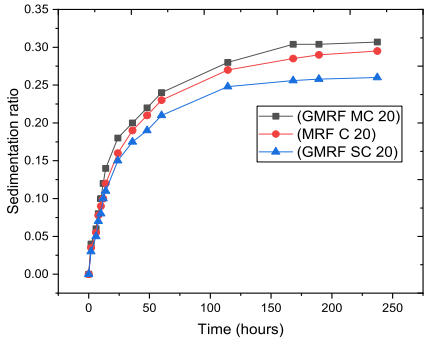

Figure 5: Sedimentation stability vs time in hours for GMRFs and MRFs (CI based).

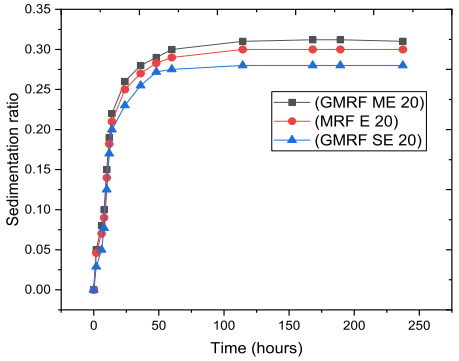

Figure 6: Sedimentation stability vs time in hours for GMRFs and MRFs (EI based).

4.2 Effect of the type of suspended particles

Different kinds of suspended particles can be used in MR fluids. The suspended particles are easily magnetizable particles. Because of this reason, iron particles are generally used as the suspended particles in MRFs. The size of the suspended particles should be small, so as to reduce sedimentation of the particles. As a result, the sedimentation stability also depends on the type of suspended particles used. Figures 5 and 6 show the graphs of sedimentation ratio versus time in hours of MRF samples using CI particles and EI particles respectively as the suspended particles. It is seen from the graphs that for the same weight percentage of suspended particles and for the same carrier fluid, sedimentation ratio with respect to time of MRF/GMRF using carbonyl iron is comparatively less. It is because the size of the CI particles is smaller than the size of the EI particles, as well as irregular shape of EI particles. MRFs having smaller size particles tends to settle less [20]. The yield strength developed is also more in case of MRF/GMRF having CI particles as the suspended particles because of the spherical structure of CI particles [7].

4.3 Effect of sedimentation velocity

The sedimentary velocity of the samples was calculated using stokes equation (2) and are tabulated in table 5 below. The sedimentation stability of GMRF SC 20 is better than the GMRF SE 20 due to small size (6 $\times 10^{-6}$ m) of CI particles than EI particles fluids (10 $\times 10^{-6}$ m). This can be better explained by the stokes equation (2).

5. CONCLUSION

Smart material has great potential for industrial applications because of its controllability. Magnetorheological fluid, being a smart material, has got great attention in the last few decades. However, the problem is the

cost associated with the synthesis of MR fluids. This work is an effort to find an alternative to conventional MRFs.

The present work focuses on amalgamation of GMRFs using natural oils such as mahua and simarouba oil as the carrier fluids and compare with the samples of MRF prepared by silicone oil as carrier fluid. It is found that the densities of simarouba (966 Kg m^{-3}) oils are more than that of silicone oil (959 Kg m^{-3}). The sedimentation stability of MR fluids were compared with sedimentation stability of GMRFs as shown in figures (5) and (6), it is seen that the sedimentation stability of the simarouba based GMRFs is more than that of silicone oil based MRFs and mahua based GMRF. This result can be attributed partly due to the relatively lesser density difference in case of simarouba based GMRFs. Moreover, the sedimentation stability using CI particles based GMRF/ MRF samples are better than that of EI particles based GMRF/MRF. Simaroubha based GMRF with CI particles i.e. GMRF SC 20 has the least sedimentation velocity (V_{sed}) of 2.485×10^{-6} m s^{-1}. The results shows a great potential that GMRF SC 20 can be substituted for conventional silicone oil based MRF, considering better stability against sedimentation.

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