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SYNTHESIS AND CHARACTERIZATION OF NANOGREASE FOR AUTOMOTIVE WHEEL BEARING APPLICATION

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With increasing number of vehicles, the technical problems of various automotive systems have also been increased. It results in shorter life span of the automotive system and also discomfort ride of a vehicle which has direct impact on maintenance cost of the vehicle. Grease is an important element in automotive wheel bearing lubrication. Considering emerging need of high load carrying capacity and viscosity, a new kind of bearing lubricants is required. Nanogrease has potential to fulfil the emerging needs of lubrications. Now a day, there is scope and also challenge to develop new grease for specific application**.** In this paper, samples of nanogrease is synthesized & characterized for thermal, tribological, rheological properties. The results of characterization are compared with conventional greases and suitable nanogrease has been suggested for automotive wheel bearing application.

Keywords**:** Nanogrease, $CaCO_3$ nanoparticles, Fe_2O_3 nanoparticles, Nano-tribology

1. Introduction

Lubrication is one of the oldest and most useful technologies. A solid or semisolid lubricant consisting of a thickening agent (soap or other additives) in a fluid lubricant is called as grease. Grease is a semi-fluid to a solid mixture of a fluid lubricant, a thickener, and additives. In general greases contain 70% to 95 % base oil, 5% to 20% thickening agent and 0 to 20 % of additives [1]. Greases are most often used instead of fluids where a lubricant is required to maintain its original position in a mechanism, especially where opportunities for frequent relubrication may be limited or economically unjustifiable. The first modern grease was calcium soaps which are not much used anymore. Later aluminum & sodium soaps were developed that could cope with higher temperature. Today, the widest greases are based on lithium which was developed in 1940. The 2012 NLGI survey shows that today about 70% of the total grease volume consists of lithium thickened soap grease [2]. This illustrates that there is no great development in lubricating grease.

With increasing the number of vehicles, the problem with friction and wear of various systems of automotive like bearing with their noisy operation are becoming more prominent. Consequently, improved understanding and optimization of grease frictional performance can offer an important way of reducing power losses in wheel bearings. At present, the literature is missing a comprehensive investigation on the influence of grease composition on friction under condition pertinent to the wheel bearing. It results in a short life of a system and discomfort ride of a vehicle which further makes more maintenance and cost. So, there is need to focus on it and improve lubrication properties of grease.

Today, the trend is to develop grease for the specific application [1], Pena-Paras et al. developed nanogrease for metal mechanics application [3]. The vast majority of automobiles are equipped with disc brakes. Although disc brakes provide better braking performance, their use puts tremendous stress on the wheel bearing grease. Higher temperatures are generated with disc brakes than those with conventional drum brakes. So, it creates wheel bearing leakage problem. This application is most demanding for a new type of grease. Nanogrease is a new type of grease which is nothing but the addition of nanoparticles in conventional grease. Nanogrease has been rise on the recent years as a way to solve thermal rheological and tribological issues and needs. The Fig. 1 illustrates the concept of nanogrease.

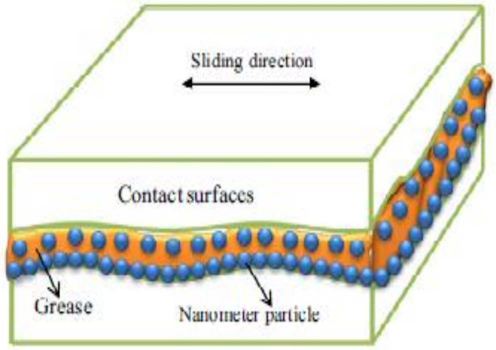

Fig. 1.1 Concept of Nanogrease [4]

Many researchers have contributed for development of general nanogrease, Xiangyu et al. [5] synthesized nanogreases using TiO_2 and SiO_2 nanoparticles and examined tribological properties and claimed that grease with TiO_2 particles exhibit good tribological characteristics; and the greases prepared using SiO_2 nano particles show good friction-reducing characteristic. Chang et al. [6] improved the lubricating performance of grease by letting tin (Sn) nanoparticles dispersed in lithium grease. Authors have concluded that then 'Sn' nanoparticles can enhance the tribological properties of lithium grease and 'Fe' nanoparticles can more effectively enhance wear resistance. Pena-Paras et al. [3] investigated that the effect of incorporating nanoparticles – TiO_2, Al_2O_3, CuO, and multi-walled carbon nanotubes (MWNTs) – within base greases like Mobilgrease 28 and Uniflor 8623B on their thermal transport and tribological properties and tribological performance. Zhao et al. [7] evaluated the friction and wear behaviour of the Nano Calcium Borate (NCB) as additive in lithium grease. Authors have found that, the addition of NCB additives in lithium grease results decrease in friction coefficient & significantly improved the anti- wear properties and load carrying capacities of the lithium grease. Xianbing et al. [8] have found that grease with $CaCO_3$ nanoparticles exhibit properties like anti-wear, friction-reduction, load-carrying capacity and extreme pressure. Also they have suggested optimum concentration of nanoparticles in grease is 5 wt. %. Shen et al. [9] developed effective nanogrease specifically for extreme operational applications. Lee et al. [10] developed nanogrease using graphite nanoparticles and evaluated tribological characteristics. Authors concluded that addition of nanoparticles decreases wear and resulted in a relatively smooth surface with fewer scars and significantly reduced metal contact.

The available literature clears that the use of nanogrease is having more advantages over conventional greases. The today's trend is to develop nanogrease for specific application; however the nanogrease is developed only for metal mechanics application. So in this research work, the different samples of nanogrease with varying concentrations of $CaCO_3$ and Fe_2O_3 nanoparticles have been synthesized by keeping view on requirement of grease for automotive wheel bearing application and characterized for thermal, tribological & rheological properties. The results of characterization have been compared with conventional greases and best suitable nanogrease has been suggested for automotive wheel bearing application.

2. Materials Selection:

In general, greases contain 70-95% of base oils, 5-20% of thickening agent, and 0-10% of additives or nanoparticles [1]. Desirable properties of grease improve not only performance of bearing but also increase the life of bearing. Stability at high temperature, anti-wear performance, resists wheel bearing leakage at high temperature, high dropping point, high water resistance and excellent cold temperature flow properties are desirable properties of grease for wheel bearing applications. Table 1 shows the desirable properties of grease for automotive wheel bearing application. By keeping view on these properties the material is selected for synthesis of nanogrease. While manufacturing the automotive application greases, the major materials need to be considered are base oil, type of nanoparticles, and type of thickener.

Table 1 Desirable properties with their required values of wheel bearing grease [11]

Properties	**Required values**	**Properties**	**Required values**
Temperature range	- 10ºC to + 170ºC	Load carrying capacity	250-340 Kg
Drop point	180-220 ºC	Penetration No.	265-295
Base oil viscosity	150º C		

The grease contains 70% to 95% base oil so that base oil plays an important role for making nanogrease for particular application. In this work, paraffin mineral oil is selected due to its good dynamic viscosity, maximum limits of impurities and having good boiling point also this oil tend to chemically mix better with soaps as well as additives and form stronger structures than naphthenic oils. Paraffinic oil has good thermal & oxidative stability also has good high temperature viscosity characteristics as compared to naphthenic oil. Table 2 shows the specifications of selected oil.

Table 2. Specifications of Paraffin Oil

Parameters	**Value**
Boiling point	300˚C at 1000hPa
Dynamic Viscosity (20˚C)	>150 mPa.s
Kinematic Viscosity (40˚C)	>34.5 CSt

Nanoparticles play several roles in lubricating grease. These primarily include enhancing the existing desirable properties, suppressing the existing undesirable properties & imparting new properties. So far, TiO_2, SiO_2, CuO, CNT (Carbon Nanotube) nanoparticles have been used in the different base greases by researchers but in this work calcium carbonate and iron oxide nanoparticles have been used due to its excellent thermo physical properties like, they can greatly improve anti-wear capability, reduce friction coefficient and increases load carrying capacity. Also these nanoparticles are chemically stable, safe for human handling and low cost compare to other nanoparticles. Table 3 shows the specifications of selected nanoparticles.

Table 3. Specifications of Nanoparticles

Parameter	**Specifications of Nanoparticles**	
Name	*Iron oxide nanoparticles*	*Calcium Carbonate nanoparticles*
Manufacturer	SIGMA ALDRICH Research Laboratories PVT. LTD	SISCO Research Laboratories PVT. LTD.
Molecular Formula	Fe_2O_3	$CaCO_3$
Molecular Wt.	159.69	100.9
Assay (purity)	Min.98%	Min.98%
Nanoparticles size	50 nm	80nm
Morphology	Cubic	Cubic
PH	8.5-9.5	8.5-9.5

3. Synthesis and Characterization of Nanogrease

The manufacturing procedure for lithium soap-based nanogrease can be split into five stages. In the first stage, the soap is formed by adding mineral oil stearic acid in to lithium hydroxide solution; hence this stage is referred to as saponification. After that dehydration will be carried out for removal of water. Then mineral oil is added to this mixture to a particular temperature. After completion of this stage, nanoparticles are added to this mixture with compensative mineral oil. Next, the grease is milled. This stage is very important because it will produce a uniform crystal and gel structure. The whole procedure is shown in Fig. 2

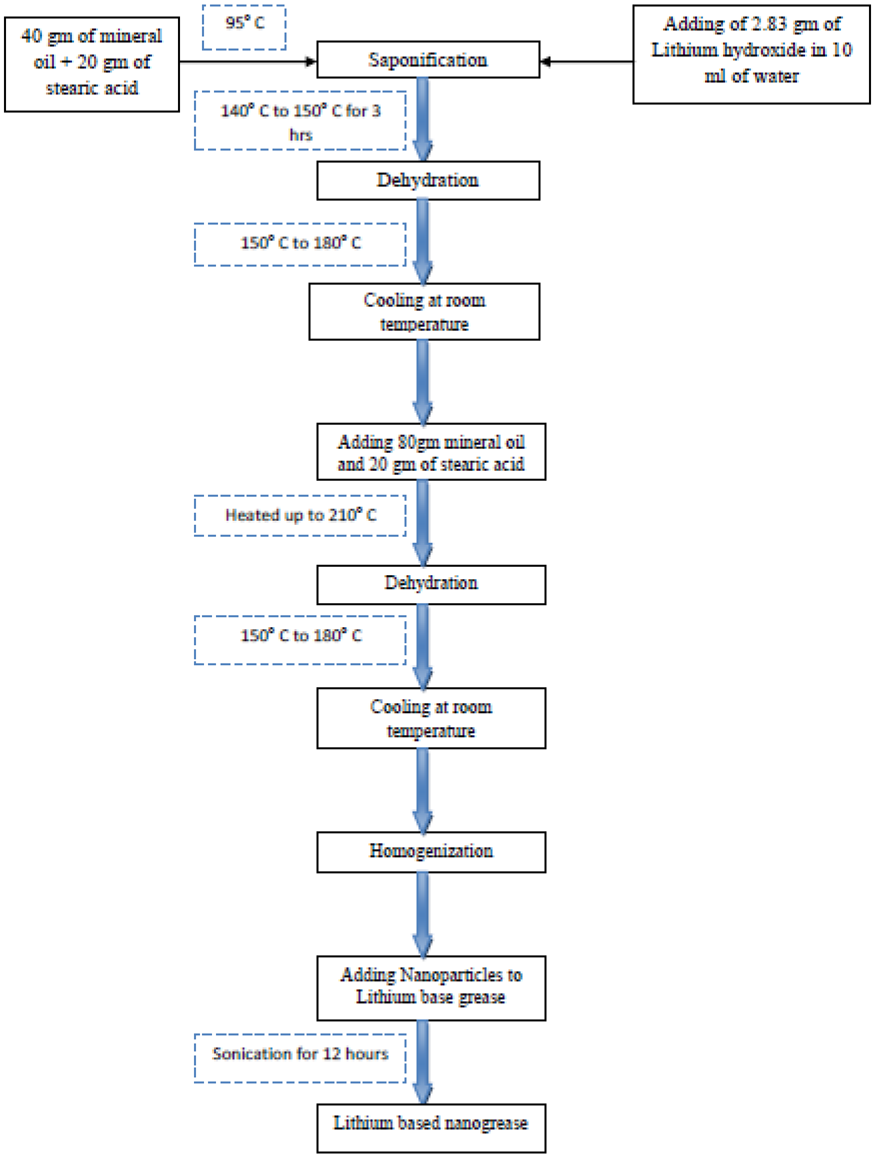

Fig. 2 Procedure for synthesis of Nanogrease [7]

The different samples of nanogrease with different weight percentage have been prepared using this procedure and some pilot characterization tests have been taken, with the help of these results nanogrease samples with 3%, 4%, 5%, 6% and 7% weight of nanoparticles have been selected for further study.

The synthesized samples of nanogrease are characterised for thermal, tribological and rheological properties. Viscosity plays important role while estimating performance of nanofluids Nield et al. [12], so that dynamic viscosity of nanogrease is measured with the help of rotational viscometer. The drop point test measures the dropping point of the lubricating greases, the dropping point is the temperature at which the greases pass from semi-solid to the liquid state under the test conditions, so it provides a practical limit of temperature above which grease cannot be used as a lubricant in the semi-solid state. So that in order to ensure the temperature limit the drop point test is conducted for the prepared grease samples. As per National Lubricating Grease Institute (NLGI) standards, penetration number gives appearance of grease through which it is clear that wether grease is soft or hard. Hence penetration test is conducted. In order to ensure load carrying capacity of grease, load carrying capacity test is carried out using four ball EP tester machine at Central Institute of Road Transport (CIRT), Pune.

4. Results and Discussions

The results carried out through different tests are analysed and plotted in order to suggest suitable grease for the automotive wheel bearing application. Fig. 3 shows the drop points of nanogrease samples with 3-7 weight precent of nanoparticles ($CaCO_3$ and Fe_2O_3).

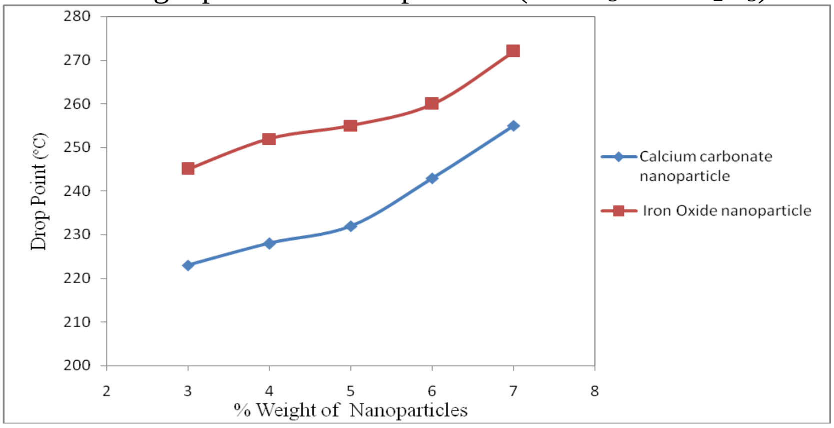

Fig. 3 Drop point of nanogrease with different percentage of nanoparticles ($CaCO_3$ and Fe_2O_3)

For wheel bearing application, the drop point temperature limit is required is 180 to 280 ºC. The drop point value of lithium base grease is 170 ºC. From the Fig. 3 it is clear that, as % weight of CaCO3 and Fe2O3 nanoparticles increases in grease, the dropping point also increases exponentially and can be achieved up to required range. The grease with a Fe2O3 nanoparticles additive shows better drop point performance than the CaCO3 nanoparticles.

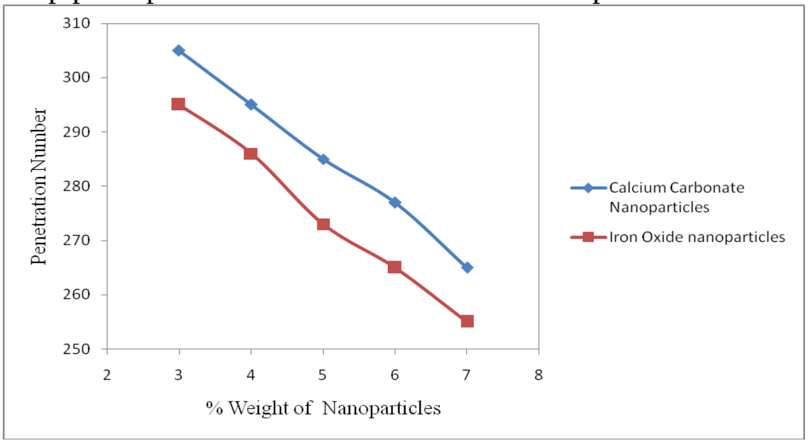

Fig. 4 Penetration number of nanogrease with different percentage of nanoparticles ($CaCO_3$ and Fe_2O_3)

Fig. 4 shows the penetration number of nanogrease samples with 3-7 weight precent of nanoparticles ($CaCO_3$ and Fe_2O_3). As lower the penetration number, the grease is harder. This means that the pumping of grease is difficult and coefficient of friction is more. However, the grease can better withstand in higher loads at lower speeds. A higher penetration number means softer grease which is having lower resistance to flow, increased pump ability but unable to withstand higher loads and more attention will have to be paid to prevent leakage. Form Fig. 4 the penetration number of Fe_2O_3 lithium grease with 7 % weight and $CaCO_3$ lithium grease with 7 % weight is 255 and 265 respectively. The penetration number or the depth of penetration was reduced to 15% and 11% for Fe_2O_3 nanoparticles and $CaCO_3$ nanoparticles respectively, if it is compared with lithium base grease which is having penetration number 350.

The load carrying capacity of lithium base grease in 170 Kg. which will be failed for wheel bearing application as it is required 250-340 Kg. The Fig. 5 shows the grease with 7 wt% of $CaCO_3$ and Fe_2O_3 exhibited better load carrying performance & could work smoothly to 340 kg and 450 kg respectively.

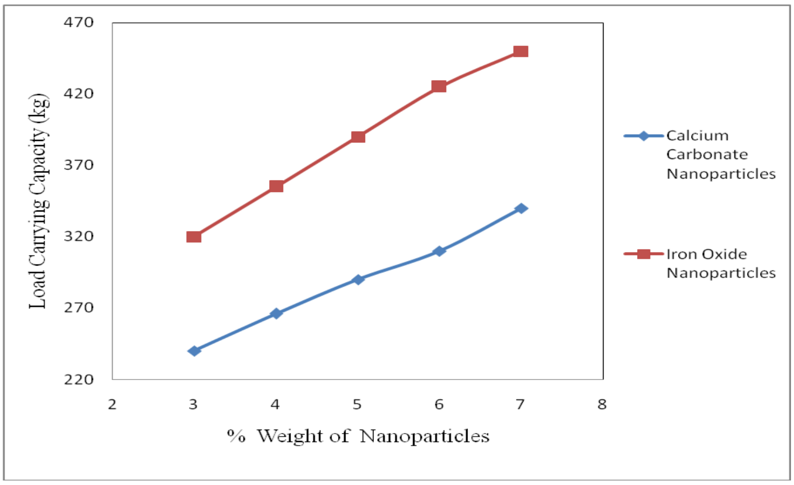

Fig. 5 Load carrying capacity of nanogrease with different percentage of nanoparticles ($CaCO_3$ and Fe_2O_3)

Fig. 6 shows that the dynamic viscosity increases with increase in nanoparticle percentage in to base grease. The grease with iron oxide particles has higher dynamic viscosity than grease with calcium carbonate nanoparticles.

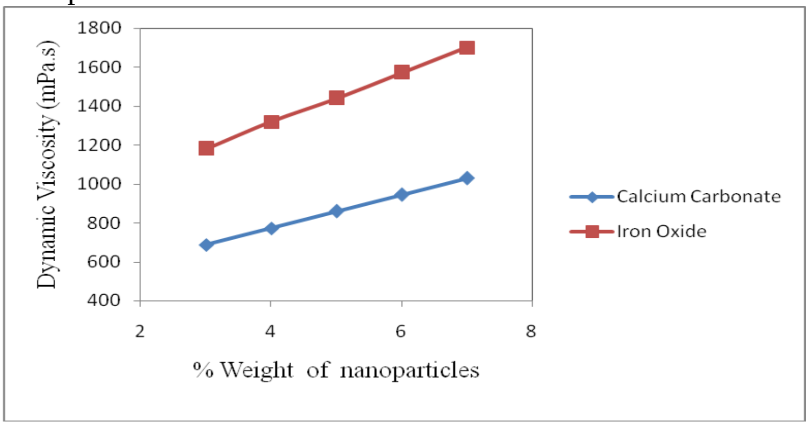

Fig. 5 Dynamic viscosity of nanogrease with different percentage of nanoparticles ($CaCO_3$ and Fe_2O_3)

5. Conclusions

In this research work, the different samples of nanogrease with varying concentrations of $CaCO_3$ and Fe_2O_3 (with 3%, 4%, 5%, 6% and 7% of weight) nanoparticles have been synthesized by keeping view on requirement of grease for automotive wheel bearing application and characterized for thermal, tribological & rheological properties like drop point, viscosity, load carrying capacity and penetration number. Experimental results showed that Fe_2O_3 and $CaCO_3$ nanoparticles can enhance the tribological and rheological properties of grease compare to base grease. Amongst these Fe_2O_3 nanoparticles can exert more excellent tribological properties as compared to $CaCO_3$ nanoparticles, when the concentration of nanoparticles is 7% weight and can significantly increase the drop point, load carrying capacity and reduce the penetration number. The sample of Fe_2O_3 nanoparticles with 7% weight increase the dropping point in drop point test by approximately 39% than the expected value. So, it is seen that in drop point test Fe_2O_3 nanoparticles shows better result than $CaCO_3$ nanoparticles. In load carrying capacity test, Fe_2O_3 nanoparticles grease sample shows 80% increment and 36% increment for $CaCO_3$ nanoparticles. In penetration test, the penetration number or the depth of penetration was reduced to 15% and 11% for Fe_2O_3 nanoparticles and $CaCO_3$ nanoparticles respectively.

Fe_2O_3 nanoparticles give better results than $CaCO_3$ nanoparticles, however the grease samples of 7% weight with Fe_2O_3 and $CaCO_3$ nanoparticles exhibit desired properties for automotive wheel bearing application. So both the samples are suitable for this application; however the cost of $CaCO_3$ nano particles are less compare to $Fe_2O_{3,}$ hence grease sample with $CaCO_3$ 7% weight can more suitable than grease sample with Fe_2O_3 nano particles. After using these samples to actual wheel bearing one can predict more insights about the performance of grease.

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