CHAPTER III

EXPERIMENTAL

3.1 Chemicals

All chemicals were obtained from various suppliers as shown in table 3.1 and they were used as received.

Table 3.1 Sources of chemicals

Number	Chemicals	Company
1	Dioctyl sebacate	Nye synthetic lubricant
2	Dioctyl adipate	Inolex chemical
3	Dioctyl azelate	Advanced Technology & Industrial Co., Ltd.
4	Irganox L57	Ciba Co, Ltd.
5	Irgamet 39	Ciba Co, Ltd.
6	STAW4	Seagate(USA) Co, Ltd.
7	Acetone	Merck
8	Ethyl alcohol	Merck
9	AK-225	Nagese
10	Potassium hydroxide	Merck
11	Mixture of toluene-isopropyl alcohol-water	Kanto chemical
12	AGH	Ridel-De-Haen
13	Silicone oil	Shinetsu, Ltd.
14	Hexane	Merck

3.2 Apparatus and Instruments

All apparatus used in this experiment are listed in table 3.2.

Table 3.2 Apparatus used in the experiment

Apparatus	Model	Manufacturer
Kinematic viscosity measurement	403-062	Rigosha&Co.,Ltd.
Flash point tester	ACO-7	Tanaka scientific Limited.
Potentiometric auto titrator	APB-410	Kyoto electronics
Karl Fischer Titrator	ADP-611/MKC-610	Kyoto electronics
Pour point tester	RPP-02CML	Rigosha & Co.,Ltd.
Density meter	35N	Anton Parr
Thermo gravity differential temperature analyzer (TG/DTA)	TG/DTA220	SEIKO instrument
Gas chromatograph / Mass spectrometer (GC/MS)	QP2010	Shimadzu
Four ball wear tester	-	Rigosha & Co.,Ltd
High-Frequency Linear-Oscillation Test Machine (SRV)	SRV III	Optimal instruments
Rotating Bomb Oxidation tester (RBOT)	RBOT-2BS	Yoshida Ltd.
Oven	DN410	Yamato scientific Co.,Ltd.
Ultrasonic	5510H	Branson
Analytical balance	GR202	AND
Microscope	SZ-PT	OLYMPUS

3.3 Sample preparation

3.3.1 Selection of ester base oils

Initially, each group of ester base oil will be considered on their kinematic viscosity as shown in table 3.3. The suitable oil should be within target range as the following explanation.

Target result

The suitable kinematic viscosity value of sample base oils should be within range of 8 – 10 mm ^{2}/s at 40 °C measuring temperature.

Table 3.3 Type of ester base oils

Ester type	Acid structure	Alcohol structure	Ester compound	Kinematic viscosity at 40 °C (unit :mm^2/s)
Monoester	Monocarboxylic	Linear	Methyl decanoate	3
		Branch	2-Ethyl hexyl oleate	29
Diester	Dicarboxylic	Linear	Dimethyl adipate	25
		Branch	Bis(2-ethyl hexyl sebacate)	12
Polyol ester	Monocarboxylic	Linear	Glycerol monooleate	90
		Branch	Glycerol trioleate	230

After investigation on the kinematic viscosity at 40 °C, the diester base oil was possible to be used in FDB spindle motor for 2.5 inch HDD. Therefore, the diester base oil was selected as potential base oil for FDB spindle motor of 2.5 inch HDD as explanation in item 3.3.2.

3.3.2 Selection of types of diester base oils

The potential candidate of diesters are as follow.

- Dioctyl adipate (DOA)
- Dioctyl azelate (DOZ)
- Dioctyl sebacate (DOS)

- Dioctyl Phthalate (DOP)
- Dioctyl terephthalate (DOTP)

The kinematic viscosity at 40 °C of DOA, DOZ and DOS oils are found to be within target range. However, the price of DOZ oil is too high. Therefore, in this research only DOA and DOS will be selected for the preparation of finished lubricant for FDB spindle motor of 2.5 inch HDD.

3.3.3 Preparation of base oil samples

The base oils were prepared by mixing DOA and DOS base oils as in table 3.4. These mixture of base oils were sonicated for 30 minutes. Finally, a completed homogeneity of mixture will be checked by visual inspection.

The mixture base oils were designated as "Sample A" to "Sample H", respectively.

Table 3.4 Percentage of DOA and DOS blending

Sample designation	% Blending	
	DOA	DOS
A	20	80
B	30	70
C	40	60
D	45	55
E	50	50
F	55	45
G	60	40
H	70	30

3.4 Properties assessment of base oil samples

The list of test methods for properties assessment of base oil samples is shown in table 3.5.

Table 3.5 Test methods for base oil samples

Test methods	ASTM no.	Appendices
Kinematic viscosity[11]	ASTM D-445	A1
Flash point [12]	ASTM D-92	A2
Total acid number [13]	ASTM D-664	A3
Moisture content [14]	ASTM D-1744-92	A4
Specific gravity [15]	ASTM D-4052	A5
Pour point [16]	ASTM D-5950	A6
Corrosive outgas test by Copper (Cu) - Silver (Ag) coupon method	-	A7
Corrosion test by Copper plate method [17]	ASTM D-130	A8
Weight loss by TG/DTA instrument	-	A9
Outgas analysis by GC/MS instrument	-	A10
Wear analysis by Four ball wear tester [19]	ASTM D-2266	A11
Friction coefficient analysis by SRV [20]	ASTM D-5707	A12
Oxidation induction time by RBOT [21]	ASTM D-2272	A13

3.5 Preparation of finished lubricant samples and properties assessment

3.5.1 Finished lubricants preparation

Base oils were blended with several additives as shown in table 3.6. These finished lubricants were sonicated for 30 minutes. Finally, a completed homogeneity of mixture would be checked by visual inspection.

Table 3.6 Type and percentage of used additives

Type of additives	Function	% Blending in base oil
Antioxidant	Resisting oxidation reaction, bearing lubricants help in preventing damage and also protect the bearings from the corrosive effects of harmful chemicals.	1%
Antiwear	To prevent metal-to-metal contact between contacting parts.	0.5%
Metal deactivator	To form protective film on running surfaces to inhibit corrosion reactions	0.05%

3.5.2 Properties assessment for finished lubricants

In this step, the properties of finished lubricants were assessed by test methods as shown in table 3.5. Moreover, evaporation loss testing, compatibility testing and service life test *via* motor running were additional testing for finished lubricant's properties assessment which were described as in table 3.7.

Table 3.7 Additional test methods for finished lubricants

Test methods	ASTM no.	Appendices
Evaporation loss	-	A14
Compatibility test between lubricants and bearing part	-	A15
Service life test	-	A16