

CHAPTER V

RESULTS AND DISCUSSIONS

5.1 General

In lubricating oil blending process, several types of base lubricating oil, having different viscosities, and additives were mixed together to obtain the homogeneity of the lubricating oils. The important factors which are used for the criteria of a suitable indicator that represents a homogenous conditions are the total volume of samples should be minimized and performed with a minimal effect on the lubricant formulation.

Among quality control items (viscosity, density, foam test, pour point and additive element), it is found that only ml. of sample size used in additive element test (minimal effect on the lubricant formulation) and the main common component in additive is Ca-compound, so Ca-content was selected as the parameter in determining the additive element. Preliminary mixing to determine variation of tests viscosity and Ca-content are examined at various times. The results showed that viscosity of the mixing oil its final expected value (within limits or specifications) in a shorter time, as shown in Table 5.1, whereas Ca-content value reached its final expected value (within limits or specifications) in a longer time.

Table 5.1 Physical properties of lubricating oil at various time

Properties	15 Sec.	60 Sec.	120 Sec.	360 Sec.	480 Sec.
Viscosity @ 100 °C, cSt	11.82	11.82	11.83	11.84	11.83
Ca-content, % Wt.	0.082	0.1125	0.1846	0.9135	0.9142

Uniformity of Ca-content was selected as the parameter to determine the required mixing time and indicate the homogeneity of the lubricating oil.

A set of 27 mixing experiment was conducted in three agitated batch mixer. In each batch mixer, different measurements are carried out to determine the required mixing time in an agitated batch mixer for the following conditions:-

1. Diameter of tank: 12 cm., 25 cm. and 36 cm.
2. Speed of impeller: 300, 400 and 500 rpm.
3. Viscosity of finish lubricating oils @ 100 °C: 12, 14 and 19 cSt.
4. Volume of lubricating oils: 1.35, 12.27 and 36.66 lts.

The above conditions were summarized in Table 5.2

Table 5.2 Experimental Conditions

Experiment No.	Viscosity of lubricating oil @ 100 Deg C, cSt. (approximate)	Diameter of tank, cm	speed, rpm.
1	12	12	300
2	12	12	400
3	12	12	500
4	12	25	300
5	12	25	400
6	12	25	500
7	12	36	300
8	12	36	400
9	12	36	500
10	14	12	300
11	14	12	400
12	14	12	500
13	14	25	300
14	14	25	400
15	14	25	500
16	14	36	300
17	14	36	400
18	14	36	500
19	19	12	300
20	19	12	400
21	19	12	500
22	19	25	300
23	19	25	400
24	19	25	500
25	19	36	300
26	19	36	400
27	19	36	500

5.2 Instrumental and Experimental error

5.2.1 Instrumental error

ICP-OES instrument is used to determine concentration of additive elements in lubricating oil following ASTM D 4951. Accuracy of the instrument is found by analysis of same sample twice. It is found that the accuracy is within ± 10 percent, as shown in Table 5.3.

5.2.2 Experimental error

Accuracy experimental method is also found by conducting the experiment at same conditions for three times. It is found that the accuracy is within ± 20 percent, as shown in Table 5.4.

Table 5.3 Precision of concentration of Ca-content at various time

Sampling time	Concentration of Ca (% Wt)							
	Instrumental error (1)				Instrumental error (2)			
	Test-1	Test-2	Average	% Error	Test-1	Test-2	Average	% Error
0.00	0.0723	0.0663	0.0693 ± 0.0030	± 8.66	0.0651	0.0665	0.0658 ± 0.0007	± 2.13
0.25	0.0695	0.0755	0.0725 ± 0.0030	± 8.28	0.0715	0.0737	0.0726 ± 0.0011	± 3.03
0.50	0.0803	0.0849	0.0826 ± 0.0023	± 5.57	0.0875	0.0883	0.0879 ± 0.0004	± 0.91
0.75	0.1052	0.1110	0.1081 ± 0.0029	± 5.37	0.1124	0.1184	0.1154 ± 0.0030	± 5.20
1.00	0.0762	0.0766	0.0764 ± 0.0002	± 0.52	0.0857	0.0883	0.0870 ± 0.0013	± 2.99
1.25	0.0846	0.0904	0.0875 ± 0.0029	± 6.63	0.0759	0.0745	0.0752 ± 0.0007	± 1.86
1.50	0.0955	0.0897	0.0926 ± 0.0029	± 6.26	0.0958	0.0950	0.0954 ± 0.0004	± 0.84
1.75	0.0958	0.1010	0.0984 ± 0.0026	± 5.28	0.1254	0.1228	0.1241 ± 0.0013	± 2.10
2.00	0.1002	0.1048	0.1025 ± 0.0023	± 4.49	0.1247	0.1243	0.1245 ± 0.0002	± 0.32
2.25	0.1254	0.1258	0.1256 ± 0.0002	± 0.32	0.1255	0.1157	0.1206 ± 0.0049	± 8.13
2.50	0.1544	0.1584	0.1564 ± 0.0020	± 2.56	0.1474	0.1436	0.1455 ± 0.0019	± 2.61
2.75	0.1254	0.1208	0.1231 ± 0.0023	± 3.74	0.1465	0.1531	0.1498 ± 0.0033	± 4.41
3.00	0.1985	0.1809	0.1897 ± 0.0088	± 9.28	0.1890	0.1809	0.1850 ± 0.0041	± 4.38
3.25	0.3022	0.3026	0.3024 ± 0.0002	± 0.13	0.2389	0.2522	0.2456 ± 0.0066	± 5.42
3.50	0.2566	0.2350	0.2458 ± 0.0108	± 8.79	0.2566	0.2522	0.2544 ± 0.0022	± 1.73
3.75	0.3687	0.4043	0.3865 ± 0.0178	± 9.21	0.3326	0.3104	0.3215 ± 0.0111	± 6.91
4.00	0.3952	0.3956	0.3954 ± 0.0002	± 0.10	0.3952	0.3938	0.3945 ± 0.0007	± 0.35
4.25	0.4233	0.4289	0.4261 ± 0.0028	± 1.31	0.4458	0.4584	0.4521 ± 0.0063	± 2.79
4.50	0.4652	0.4484	0.4568 ± 0.0084	± 3.68	0.4985	0.5063	0.5024 ± 0.0039	± 1.55
4.75	0.5215	0.5173	0.5194 ± 0.0021	± 0.81	0.4325	0.4105	0.4215 ± 0.0110	± 5.22
5.00	0.5587	0.5537	0.5562 ± 0.0025	± 0.90	0.5120	0.5308	0.5214 ± 0.0094	± 3.61
5.25	0.6425	0.5823	0.6124 ± 0.0301	± 9.83	0.5847	0.5947	0.5897 ± 0.0050	± 1.70
5.50	0.7355	0.7373	0.7364 ± 0.0009	± 0.24	0.8654	0.8440	0.8547 ± 0.0107	± 2.50
5.75	0.7586	0.8116	0.7851 ± 0.0265	± 6.75	0.8657	0.8853	0.8755 ± 0.0098	± 2.24
6.00	0.6542	0.6160	0.6351 ± 0.0191	± 6.01	0.8900	0.8826	0.8863 ± 0.0037	± 0.83
6.25	0.8245	0.8405	0.8325 ± 0.0080	± 1.92	0.9145	0.9103	0.9124 ± 0.0021	± 0.46
6.50	0.8569	0.9021	0.8795 ± 0.0226	± 5.14	0.9458	0.9590	0.9524 ± 0.0066	± 1.39
6.75	0.8012	0.7896	0.7954 ± 0.0058	± 1.46	0.9450	0.9646	0.9548 ± 0.0098	± 2.05
7.00	0.8857	0.8873	0.8865 ± 0.0008	± 0.18	0.9542	0.9308	0.9425 ± 0.0117	± 2.48
7.25	0.9048	0.9056	0.9052 ± 0.0004	± 0.09	0.9456	0.9260	0.9358 ± 0.0098	± 2.09

Table 5.3 Precision of concentration of Ca-content at various time (Continued)

Sampling time	Concentration of Ca (% Wt)							
	Instrumental error (1)				Instrumental error (2)			
	Test-1	Test-2	Average	% Error	Test-1	Test-2	Average	% Error
7.50	0.9245	0.9403	0.9324 ± 0.0079	± 1.69	0.9245	0.9175	0.9210 ± 0.0035	± 0.76
7.75	0.9458	0.9618	0.9538 ± 0.0080	± 1.68	0.9247	0.9249	0.9248 ± 0.0001	± 0.02
8.00	0.9910	1.0192	1.0051 ± 0.0141	± 2.81	0.9145	0.8887	0.9016 ± 0.0129	± 2.86
8.25	0.9980	0.9662	0.9821 ± 0.0159	± 3.24	0.9245	0.9471	0.9358 ± 0.0113	± 2.42
8.50	0.9350	0.9554	0.9452 ± 0.0102	± 2.16	0.8567	0.9363	0.8965 ± 0.0398	± 8.88
8.75	0.8955	0.8987	0.8971 ± 0.0016	± 0.36	0.9180	0.8890	0.9035 ± 0.0145	± 3.21
9.00	0.8874	0.8212	0.8543 ± 0.0331	± 7.75	0.9015	0.9019	0.9017 ± 0.0002	± 0.04
9.25	0.8852	0.8834	0.8843 ± 0.0009	± 0.20	0.9120	0.9016	0.9068 ± 0.0052	± 1.15
9.50	0.8659	0.9223	0.8941 ± 0.0282	± 6.31	0.8999	0.9031	0.9015 ± 0.0016	± 0.35
9.75	0.9015	0.9075	0.9045 ± 0.0030	± 0.66	0.9015	0.9010	0.9013 ± 0.0002	± 0.06
10.00	0.8745	0.8941	0.8843 ± 0.0098	± 2.22	0.9150	0.8920	0.9035 ± 0.0115	± 2.55
10.25	0.8666	0.8684	0.8675 ± 0.0009	± 0.21	0.9032	0.9036	0.9034 ± 0.0002	± 0.04
10.50	0.8569	0.9381	0.8975 ± 0.0406	± 9.05	0.8978	0.9158	0.9068 ± 0.0090	± 1.99
10.75	0.8877	0.8813	0.8845 ± 0.0032	± 0.72	0.9160	0.8970	0.9065 ± 0.0095	± 2.10
11.00	0.8842	0.8762	0.8802 ± 0.0040	± 0.91	0.9014	0.9150	0.9082 ± 0.0068	± 1.50
11.25	0.8755	0.8743	0.8749 ± 0.0006	± 0.14	0.9210	0.8926	0.9068 ± 0.0142	± 3.13
11.50	0.8566	0.8730	0.8648 ± 0.0082	± 1.90	0.9124	0.8972	0.9048 ± 0.0076	± 1.68
11.75	0.8799	0.8977	0.8888 ± 0.0089	± 2.00	0.9047	0.8977	0.9012 ± 0.0035	± 0.78
12.00	0.8999	0.8803	0.8901 ± 0.0098	± 2.20	0.9034	0.9042	0.9038 ± 0.0004	± 0.09
12.25	0.9011	0.9013	0.9012 ± 0.0001	± 0.02	0.8950	0.9050	0.9000 ± 0.0050	± 1.11
12.50	0.8799	0.8913	0.8856 ± 0.0057	± 1.29	0.9017	0.9011	0.9014 ± 0.0003	± 0.07
12.75	0.8578	0.8360	0.8469 ± 0.0109	± 2.57	0.9064	0.9066	0.9065 ± 0.0001	± 0.02
13.00	0.8459	0.9059	0.8759 ± 0.0300	± 6.85	0.9035	0.9059	0.9047 ± 0.0012	± 0.27
13.25	0.8547	0.8743	0.8645 ± 0.0098	± 2.27	0.9102	0.9034	0.9068 ± 0.0034	± 0.75
13.50	0.8888	0.8208	0.8548 ± 0.0340	± 7.96	0.9120	0.8948	0.9034 ± 0.0086	± 1.90
13.75	0.8846	0.8848	0.8847 ± 0.0001	± 0.02	0.9047	0.9067	0.9057 ± 0.0010	± 0.22
14.00	0.8852	0.8946	0.8899 ± 0.0047	± 1.06	0.9124	0.9006	0.9065 ± 0.0059	± 1.30
14.25	0.8862	0.8872	0.8867 ± 0.0005	± 0.11	0.9145	0.8883	0.9014 ± 0.0131	± 2.91
14.50	0.8875	0.8877	0.8876 ± 0.0001	± 0.02	0.9014	0.9160	0.9087 ± 0.0073	± 1.61
14.75	0.8854	0.8864	0.8859 ± 0.0005	± 0.11	0.8854	0.8745	0.8800 ± 0.0054	± 1.24

Table 5.3 Precision of concentration of Ca-content at various time (Continued)

Sampling time	Concentration of Ca (% Wt)							
	Instrumental error (1)				Instrumental error (2)			
	Test-1	Test-2	Average	% Error	Test-1	Test-2	Average	% Error
15.00	0.8579	0.9129	0.8854 ± 0.0275	± 6.21	0.8970	0.9146	0.9058 ± 0.0088	± 1.94
16.00	0.8856	0.8892	0.8874 ± 0.0018	± 0.41	0.9045	0.9153	0.9099 ± 0.0054	± 1.19
17.00	0.8765	0.8373	0.8569 ± 0.0196	± 4.57	0.9158	0.9049	0.9104 ± 0.0054	± 1.20
18.00	0.8799	0.9151	0.8975 ± 0.0176	± 3.92	0.9140	0.9014	0.9077 ± 0.0063	± 1.39
19.00	0.8755	0.8757	0.8756 ± 0.0001	± 0.02	0.9125	0.9008	0.9067 ± 0.0058	± 1.29
20.00	0.8569	0.9123	0.8846 ± 0.0277	± 6.26	0.9040	0.9056	0.9048 ± 0.0008	± 0.18
25.00	0.8857	0.8925	0.8891 ± 0.0034	± 0.76	0.9024	0.9092	0.9058 ± 0.0034	± 0.75
30.00	0.8879	0.9051	0.8965 ± 0.0086	± 1.92	0.9024	0.9066	0.9045 ± 0.0021	± 0.46
35.00	0.8548	0.8960	0.8754 ± 0.0206	± 4.71	0.8970	0.9050	0.9010 ± 0.0040	± 0.89
40.00	0.8857	0.8785	0.8821 ± 0.0036	± 0.82	0.9040	0.9056	0.9048 ± 0.0008	± 0.18
45.00	0.8657	0.9145	0.8901 ± 0.0244	± 5.48	0.9410	0.9690	0.9550 ± 0.0140	± 2.93
50.00	0.8456	0.9298	0.8877 ± 0.0421	± 9.49	0.9102	0.8936	0.9019 ± 0.0083	± 1.84
55.00	0.8542	0.9368	0.8955 ± 0.0413	± 9.22	0.9140	0.8948	0.9044 ± 0.0096	± 2.12
60.00	0.8954	0.8688	0.8821 ± 0.0133	± 3.02	0.9050	0.9066	0.9058 ± 0.0008	± 0.18

Table 5.4 Variation of Ca-content at various time

Sampling time	Concentration of Ca (% Wt)									
	Experimental error (1)					Experimental error (2)				
	batch1	batch2	batch3	Average	% Error	batch1	batch2	batch3	Average	% Error
0.00	0.0693	0.0758	0.0658	0.0703 ± 0.0033	± 14.22	0.0543	0.0621	0.0584	0.0583 ± 0.0026	± 13.39
0.25	0.0725	0.0698	0.0726	0.0716 ± 0.0009	± 3.91	0.0745	0.0692	0.0726	0.0721 ± 0.0018	± 7.35
0.50	0.0826	0.0956	0.0879	0.0887 ± 0.0043	± 14.66	0.0987	0.0897	0.0879	0.0921 ± 0.0036	± 11.73
0.75	0.1081	0.1068	0.1154	0.1101 ± 0.0029	± 7.81	0.0951	0.0845	0.0865	0.0887 ± 0.0035	± 11.95
1.00	0.0764	0.0752	0.0875	0.0797 ± 0.0041	± 15.43	0.0675	0.0654	0.0758	0.0696 ± 0.0035	± 14.95
1.25	0.0875	0.0921	0.0752	0.0849 ± 0.0056	± 19.90	0.0989	0.0925	0.0875	0.0930 ± 0.0038	± 12.26
1.50	0.0926	0.0855	0.0954	0.0912 ± 0.0033	± 10.86	0.1095	0.0957	0.0954	0.1002 ± 0.0047	± 14.07
1.75	0.0984	0.0989	0.1145	0.1039 ± 0.0054	± 15.49	0.1645	0.1457	0.1587	0.1563 ± 0.0063	± 12.03
2.00	0.1025	0.1125	0.1245	0.1132 ± 0.0073	± 19.44	0.1742	0.1784	0.1568	0.1698 ± 0.0072	± 12.72
2.25	0.1256	0.1125	0.1201	0.1194 ± 0.0044	± 10.97	0.2174	0.2256	0.1985	0.2138 ± 0.0090	± 12.67
2.50	0.1564	0.1358	0.1455	0.1459 ± 0.0069	± 14.12	0.2945	0.3124	0.3015	0.3028 ± 0.0060	± 5.91
2.75	0.1231	0.1145	0.1345	0.1240 ± 0.0067	± 16.12	0.2854	0.2586	0.2874	0.2771 ± 0.0096	± 10.39
3.00	0.1897	0.1568	0.1847	0.1771 ± 0.0110	± 18.58	0.3274	0.3321	0.3124	0.3240 ± 0.0066	± 6.08
3.25	0.3024	0.2586	0.2658	0.2756 ± 0.0146	± 15.89	0.3551	0.3547	0.3457	0.3518 ± 0.0031	± 2.67
3.50	0.2458	0.2654	0.2544	0.2552 ± 0.0065	± 7.68	0.4035	0.3958	0.3547	0.3847 ± 0.0163	± 12.89
3.75	0.3865	0.3657	0.3215	0.3579 ± 0.0217	± 18.16	0.4558	0.4874	0.4215	0.4549 ± 0.0220	± 14.49
4.00	0.3954	0.4125	0.3945	0.4008 ± 0.0060	± 4.49	0.5647	0.5748	0.5897	0.5764 ± 0.0083	± 4.34
4.25	0.4261	0.4452	0.4521	0.4411 ± 0.0087	± 5.89	0.4965	0.5015	0.5514	0.5165 ± 0.0183	± 10.63
4.50	0.4568	0.4875	0.5024	0.4822 ± 0.0152	± 9.46	0.6421	0.6658	0.6324	0.6468 ± 0.0111	± 5.16
4.75	0.5194	0.5021	0.5215	0.5143 ± 0.0065	± 3.77	0.7135	0.7524	0.7245	0.7301 ± 0.0130	± 5.33
5.00	0.5562	0.4958	0.5214	0.5245 ± 0.0201	± 11.52	0.8641	0.8957	0.8475	0.8691 ± 0.0161	± 5.55
5.25	0.6124	0.5698	0.5897	0.5906 ± 0.0142	± 7.21	0.9532	0.9254	0.9854	0.9547 ± 0.0200	± 6.28
5.50	0.7364	0.7564	0.8547	0.7825 ± 0.0394	± 15.12	0.9881	0.9874	0.9587	0.9781 ± 0.0098	± 3.01
5.75	0.7851	0.8457	0.8755	0.8354 ± 0.0301	± 10.82	0.9673	0.9647	0.9744	0.9688 ± 0.0032	± 1.00
6.00	0.8351	0.8654	0.8864	0.8623 ± 0.0171	± 5.95	0.9321	0.9861	0.8864	0.9349 ± 0.0332	± 10.66
6.25	0.8325	0.8659	0.9124	0.8703 ± 0.0266	± 9.18	0.8943	1.0250	0.9124	0.9439 ± 0.0436	± 13.85
6.50	0.8795	0.9254	0.9524	0.9191 ± 0.0243	± 7.93	0.9245	0.9845	0.9524	0.9538 ± 0.0200	± 6.29
6.75	0.7954	0.9564	0.9548	0.9022 ± 0.0537	± 17.85	0.9041	0.9726	0.9548	0.9438 ± 0.0228	± 7.26
7.00	0.8865	0.9875	0.9425	0.9388 ± 0.0337	± 10.76	0.9086	0.9125	0.9425	0.9212 ± 0.0113	± 3.68
7.25	0.9052	0.9579	0.9358	0.9330 ± 0.0176	± 5.65	0.9143	0.9458	0.9358	0.9320 ± 0.0105	± 3.38

Table 5.4 Variation of Ca-content at various time (Continued)

Sampling time	Concentration of Ca (% Wt)									
	Experimental error (1)					Experimental error (2)				
	batch1	batch2	batch3	Average	% Error	batch1	batch2	batch3	Average	% Error
7.50	0.9324	0.9589	0.9210	0.9374 ± 0.0126	± 4.04	0.9042	0.9321	0.9210	0.9191 ± 0.0093	± 3.04
7.75	0.9538	0.9958	0.9248	0.9581 ± 0.0237	± 7.41	0.9158	0.8952	0.9248	0.9119 ± 0.0099	± 3.25
8.00	1.0051	0.9758	0.9016	0.9608 ± 0.0345	± 10.77	0.9109	0.9142	0.9016	0.9089 ± 0.0042	± 1.39
8.25	0.9821	0.9658	0.9358	0.9612 ± 0.0154	± 4.82	0.9002	0.9012	0.9358	0.9124 ± 0.0119	± 3.90
8.50	0.9452	0.9578	0.8965	0.9332 ± 0.0204	± 6.57	0.8945	0.9014	0.8965	0.8975 ± 0.0023	± 0.77
8.75	0.8971	0.9245	0.9035	0.9084 ± 0.0091	± 3.02	0.9045	0.9019	0.9035	0.9033 ± 0.0009	± 0.29
9.00	0.8543	0.9074	0.9017	0.8878 ± 0.0177	± 5.98	0.9106	0.9011	0.9017	0.9045 ± 0.0032	± 1.05
9.25	0.8843	0.9168	0.9068	0.9026 ± 0.0108	± 3.60	0.9004	0.9021	0.9068	0.9031 ± 0.0021	± 0.71
9.50	0.8941	0.9158	0.9015	0.9038 ± 0.0072	± 2.40	0.9058	0.9018	0.9015	0.9030 ± 0.0014	± 0.48
9.75	0.9045	0.9087	0.9015	0.9049 ± 0.0024	± 0.80	0.8990	0.9015	0.9015	0.9007 ± 0.0008	± 0.28
10.00	0.8843	0.9164	0.9035	0.9014 ± 0.0107	± 3.56	0.9103	0.9015	0.9035	0.9051 ± 0.0029	± 0.97
10.25	0.8675	0.9147	0.9034	0.8952 ± 0.0157	± 5.27	0.9016	0.9055	0.9034	0.9035 ± 0.0013	± 0.43
10.50	0.8975	0.9168	0.9068	0.9070 ± 0.0064	± 2.13	0.9018	0.9078	0.9068	0.9055 ± 0.0020	± 0.66
10.75	0.8845	0.9099	0.9065	0.9003 ± 0.0085	± 2.82	0.9058	0.9105	0.9065	0.9076 ± 0.0016	± 0.52
11.00	0.8802	0.9154	0.9082	0.9013 ± 0.0117	± 3.91	0.9088	0.9018	0.9082	0.9063 ± 0.0023	± 0.77
11.25	0.8749	0.9175	0.9068	0.8997 ± 0.0142	± 4.73	0.9099	0.9011	0.9068	0.9059 ± 0.0029	± 0.97
11.50	0.8648	0.9121	0.9048	0.8939 ± 0.0158	± 5.29	0.9028	0.8925	0.9048	0.9000 ± 0.0041	± 1.37
11.75	0.8888	0.9132	0.9012	0.9011 ± 0.0081	± 2.71	0.8988	0.9088	0.9012	0.9029 ± 0.0033	± 1.11
12.00	0.8901	0.9164	0.9038	0.9034 ± 0.0088	± 2.91	0.8836	0.9031	0.9038	0.8968 ± 0.0067	± 2.25
12.25	0.9012	0.9158	0.9000	0.9057 ± 0.0053	± 1.74	0.9106	0.9049	0.9000	0.9052 ± 0.0035	± 1.17
12.50	0.8856	0.9146	0.9014	0.9005 ± 0.0097	± 3.22	0.9201	0.9106	0.9014	0.9107 ± 0.0062	± 2.05
12.75	0.8469	0.9099	0.9065	0.8878 ± 0.0210	± 7.10	0.9036	0.9088	0.9065	0.9063 ± 0.0017	± 0.57
13.00	0.8759	0.9088	0.9047	0.8965 ± 0.0110	± 3.67	0.9089	0.9101	0.9047	0.9079 ± 0.0018	± 0.59
13.25	0.8645	0.9187	0.9068	0.8967 ± 0.0181	± 6.04	0.9048	0.9088	0.9068	0.9068 ± 0.0013	± 0.44
13.50	0.8548	0.9089	0.9034	0.8890 ± 0.0180	± 6.09	0.9045	0.9038	0.9034	0.9039 ± 0.0004	± 0.12
13.75	0.8847	0.9124	0.9057	0.9009 ± 0.0092	± 3.07	0.9047	0.8991	0.9057	0.9032 ± 0.0022	± 0.73
14.00	0.8899	0.9177	0.9065	0.9047 ± 0.0093	± 3.07	0.9089	0.9011	0.9065	0.9055 ± 0.0026	± 0.86
14.25	0.8867	0.9168	0.9014	0.9016 ± 0.0100	± 3.34	0.9099	0.9033	0.9014	0.9049 ± 0.0028	± 0.94
14.50	0.8876	0.9110	0.9087	0.9024 ± 0.0078	± 2.59	0.9016	0.9019	0.9087	0.9041 ± 0.0024	± 0.79
14.75	0.8859	0.9145	0.9035	0.9013 ± 0.0095	± 3.17	0.8999	0.8979	0.9035	0.9004 ± 0.0019	± 0.62

Table 5.4 Variation of Ca-content at various time (Continued)

Sampling time	Concentration of Ca (% Wt)									
	Experimental error (1)					Experimental error (2)				
	batch1	batch2	batch3	Average	% Error	batch1	batch2	batch3	Average	% Error
15.00	0.8854	0.9199	0.9058	0.9037 ± 0.0115	± 3.82	0.9059	0.9033	0.9058	0.9050 ± 0.0009	± 0.29
16.00	0.8874	0.9124	0.9099	0.9032 ± 0.0083	± 2.77	0.9106	0.9015	0.9099	0.9073 ± 0.0030	± 1.00
17.00	0.8569	0.9144	0.9101	0.8938 ± 0.0192	± 6.43	0.9011	0.9014	0.9101	0.9042 ± 0.0030	± 1.00
18.00	0.8975	0.9156	0.9077	0.9069 ± 0.0060	± 2.00	0.9111	0.9011	0.9077	0.9066 ± 0.0033	± 1.10
19.00	0.8756	0.9140	0.9065	0.8987 ± 0.0128	± 4.27	0.9135	0.9045	0.9065	0.9082 ± 0.0030	± 0.99
20.00	0.8846	0.9120	0.9048	0.9005 ± 0.0091	± 3.04	0.9112	0.9035	0.9048	0.9065 ± 0.0026	± 0.85
25.00	0.8891	0.9111	0.9058	0.9020 ± 0.0073	± 2.44	0.9016	0.9048	0.9058	0.9041 ± 0.0014	± 0.46
30.00	0.8965	0.9058	0.9045	0.9023 ± 0.0031	± 1.03	0.9089	0.9047	0.9045	0.9060 ± 0.0015	± 0.49
35.00	0.8754	0.9122	0.9010	0.8962 ± 0.0123	± 4.11	0.9077	0.9045	0.9010	0.9044 ± 0.0022	± 0.74
40.00	0.8821	0.9147	0.9048	0.9005 ± 0.0109	± 3.62	0.9083	0.9028	0.9048	0.9053 ± 0.0018	± 0.61
45.00	0.8901	0.9099	0.9550	0.9183 ± 0.0216	± 7.07	0.9078	0.9012	0.9550	0.9213 ± 0.0179	± 5.84
50.00	0.8877	0.9136	0.9019	0.9011 ± 0.0086	± 2.87	0.9025	0.9046	0.9019	0.9030 ± 0.0009	± 0.30
55.00	0.8955	0.9155	0.9044	0.9051 ± 0.0067	± 2.21	0.9054	0.9044	0.9044	0.9047 ± 0.0003	± 0.11
60.00	0.8821	0.9152	0.9058	0.9010 ± 0.0110	± 3.67	0.9135	0.9075	0.9058	0.9089 ± 0.0026	± 0.85

5.3 Determination of required mixing time

The mixing time, t_m , is defined as the time required to reach a specified level of uniformity in a mixing system.

In order to obtain the Ca-content from the experimental data. The results are presented as a plot of time of sampling versus the Ca-content as shown in Figure 5.1.

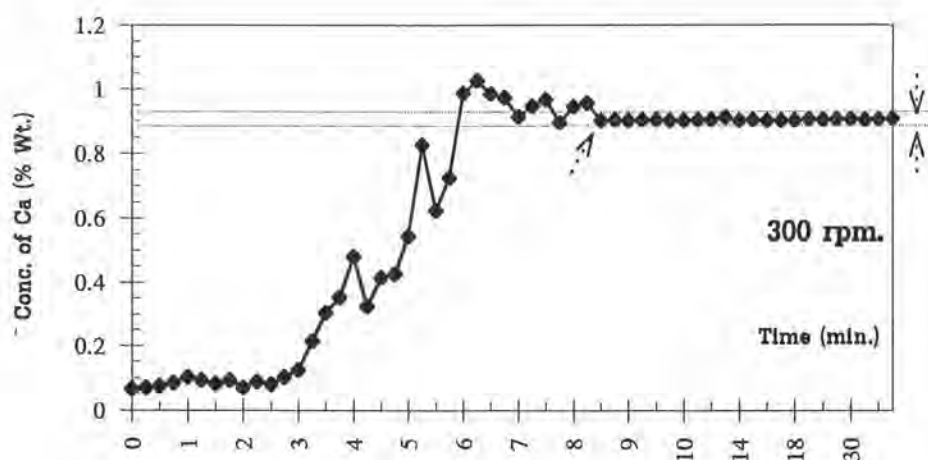


Figure 5.1 Mixing curve of Ca-content versus time of sampling

From this figure, only one datum was obtained at each measuring interval and this datum was the required mixing time. From visual examination, it was observed that the homogeneous solution was obtained after the Ca-content reached the steady mean value. This was done by comparison with the standard deviation. Their standard deviation is significantly constant to represent the condition of homogeneous lubricating oil.

AICHe Equipment Testing Procedure (Dry Solid, Paste and Dough Mixing Equipment, 1979) recommended the selection of the mixing time of the curing-rate additive as the time required to achieve concentration of mixture within acceptable limit or to reach composition equilibrium by statistical analyzing.

Pipop T. (1992) studied the mixing of water and concentration of NaCl solutions using the turbines and tracer technique both the radioactive and conductivity methods. The time of mixing, t_m , was selected as the time to achieve variation of concentration of radioactive material to become less than ± 3 times of standard deviation as shown in Figure 3.11.

In this study, it was found that the distribution data of Ca-content gave the results in the manner similar to tracer response curve by Pipop T. So, the

time to achieve variation of concentration of mixture from their mean within ± 3 times of their standard deviation was the proper condition to determine the required mixing time.

For sample calculation, from Figure 5.1 as mixing time curve of a experiment (Viscosity of lubricating oil @ 100 °C \approx 12 cSt, 12 cm of diameter tank, 300 rpm of impeller speed), by statistical analysis as shown in Appendix F, the mean and standard deviation at equilibrium were 0.9036 and 0.0036 respectively. The upper and lower limits were 0.9144 and 0.8927 respectively. The required mixing time as defined in chapter 3.10 was determined by the shortest time in which the variation of Ca-content are within those limits. The shortest time in this case is 8.5 min. the other results may be calculated with the same method as summarized in Table 5.5 and the other graphs are shown in Appendix G.

The variation of homogeneous solution may be caused by two main factors. Firstly, the nature of atomic emission (Ca) itself is randomly emitted from the element substance so the detection will never be the same value. Secondly, the limitation of sensitivity of measuring instrument must be considered.

From Table 5.5, the results were plotted between rotational speed of impeller and mixing time as shown in Figure 5.2, 5.4, 5.5.

Table 5.5 Mixing time from experiments

Viscosity of lubricating oil @ 100°C , cSt.	Diameter of tank cm.	Speed, rpm.	Mixing time, t_m Min.
12	12	300	8.5
		400	6.2
		500	4.9
	25	300	8.8
		400	6.8
		500	5.8
	36	300	9.5
		400	7.7
		500	6.4
14	12	300	8.7
		400	6.4
		500	5.0
	25	300	9.0
		400	7.0
		500	5.9
	36	300	9.8
		400	7.9
		500	6.6
19	12	300	8.9
		400	6.6
		500	5.3
	25	300	9.3
		400	7.2
		500	6.1
	36	300	10.2
		400	8.2
		500	6.9

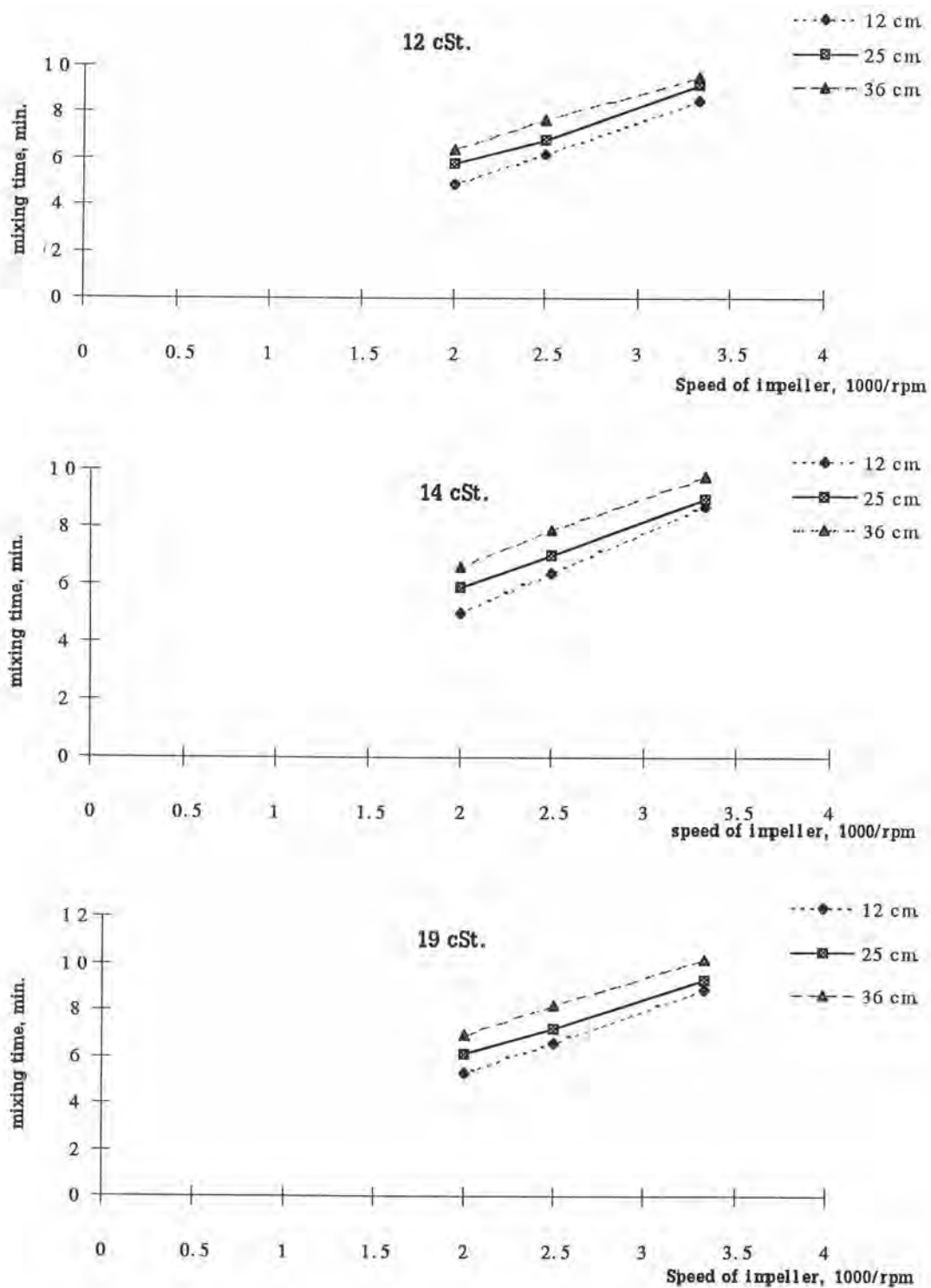


Figure 5.2 Relationship between mixing time and rotation speed

5.3.1 Effect of rotational speed of impeller

Plotted of mixing time, t_m , versus inversely rotational speed, $1/N$, were prepared on normal co-ordinates. All correlation line with a slope as shown in Table 5.6. was obtained as Figure 5.2. Before plotting, the data were corrected to the same the result of Kramers' and Marr's experimental measurement.

Kramer et al (1953) studied the mixing time of weak and concentrated KCl solutions using the propellers and turbines. Two size of vessels were used (10.5 in. and 21. in. diameter tank). Kramer used electrical conductivity cell for measuring fluctuations of solutions. The data which Kramer obtained from experiments shown that the mixing time, t_m , was found to be inversely proportional to the agitator rotation speed, N , as shown in Figure 3.12. The line shown has a slope of 6.86.

Marr (1963) made experimental measurements of batch mixing time using a phenolphthalein indicator, NaOH and HCl solution. First, NaOH and indicator were added to the vessel with the agitator in motion. Then, HCl solution was added adjacent to the impeller and the time for the red color to disappear was measured. A 11.5 in. diameter tank with three 1.5 in. wide, vertical baffles was used. The experiments were conducted by the separate effects on mixing time of each following variables: propeller diameter, D_p , rotational speed, N , depth of liquid in the tank, Z_l , and liquid viscosity, μ . It found that the results obtained the correlation:

$$t_m = f \{ Z_l, T, N, D_p, \mu, \rho \} \quad (5.1)$$

For Marr's data on mixing time correlation:

$$(t_m N)(D/T)^2 (N^2 D/g)^{1/6} (Z_l/T)^{1/2} = \text{constant} \quad (5.2)$$

For constant geometrical tank, the correlation will be changed to:

$$t_m \propto 1/N \quad (5.3)$$

From this correlation, the mixing time, t_m , was found to be inversely proportional to the agitator rotational speed, N . The data which Marr obtained on experiments are shown in Figure 3.13. The line shown has a slope of 6.1.

Figure 5.2 showed that for the same tank diameter, the required mixing time was shorter as rotational speed of impeller was increased and inversely proportion to the rotational speed in a similar manner compare with the result of Kramers' and Marr's experimental measurement. This effect of rotational speed from this study can be compared with the effect of rotational speed obtained by Kramers' and Marr's experiments as shown in Table 5.6.

Table 5.6 Comparison of the effect of rotational speed on mixing time

System of study	Tank diameter,cm	Slope, sec/speed	Reference
Lubricating oils VK.=12 cSt.	12	162.78	This study
	25	136.26	
	36	138.84	
Lubricating oils VK.=14 cSt.	12	166.80	
	25	140.28	
	36	143.70	
Lubricating oils VK.=19 cSt.	12	162.84	
	25	144.90	
	36	148.38	
KCl + water	27	6.86	Kramer
NaOH + HCl	30	6.10	Marr

A similar comparison can be made for Kramers', Marr's and this study in mixing data. The longer value of a slope of correlation line obtained by this study because of the higher difference in viscosity of components and final product.

From this experiments, at high rotation speed, the eddy diffusion will have increasing influence on the further movement of lubricating oils. The eddy diffusion depends on the return flow pattern beneath the impeller as experiment of Nunhez and McGreavy (1992) showed in Figure 5.3.

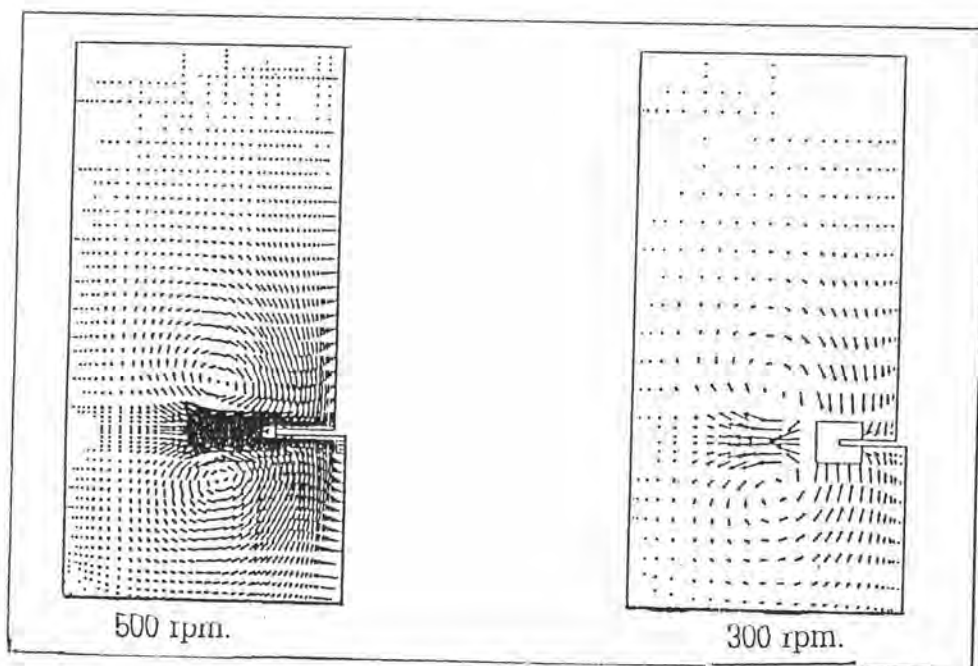


Figure 5.3 Flow patterns at different impeller speeds, (Nunhez and McGreavy, 1992)

5.3.2 Effect of tank diameter

In general, power number, N_p , as defined in chapter 3.8 was correlated with relationship:

$$N_p = P_g / \rho N^3 D_t^5 \quad (5.4)$$

And the power consumption, P_{t_m} , can be shown in relationship as below:

$$P_{t_m} = N_p t_m N^3 D_t^5 / g \quad (5.5)$$

From this equation; If dimensionless mixing time, Nt_m , was assumed to be constant and N_p also was assumed to be constant as dimensions were increased for geometrically similar equipment, the above equation can be derived to:

$$(P_{t_m} / T^3) \propto N^2 D_t^2 \quad (5.6)$$

This equation can be interpreted that the energy per unit volume is constant when the peripheral velocity of the agitator blades and density are constant in geometrically similar equipment. Under this condition, t_m is proportional to T or D_t (since Nt_m is constant)

Kramer et al included two vessel sizes in their investigation of batch mixing times. The result of Kramer showed that a slight increase in rotational speed was needed to obtain the same mixing time in large vessels.

Figure 5.4 showed that, a longer mixing time obtained by 36 cm. diameter tank than by the 12 cm. diameter tank at the same viscosity and rotational speed. This is caused from low shear rate or low velocity of flow are produced by a large-quantity of lubricating oils or by a large-diameter of tank.

From this results it should be possible to duplicate flow pattern from small-scale experiment work to large-scale equipment will become similar for scale-up on a geometrically similar basis.

5.3.3 Effect of viscosity

A plot of shear stress against shear rate was shown in Figure 3.14, for high viscosity liquids, viscosities decrease with increasing shear rate. The power relation as described in Chapter 3.8 was changed to:

$$P \propto (N^2 D_i^3 \mu) \quad (5.7)$$

In general, for constant configuration and speed

$$P \propto \mu \quad (5.8)$$

Figure 3.14, plott of apparent viscosity versus shear rate will be a straight line. For the above explanation, it can be confirmed with Metzner's and Otto's experiments.

Metzer and Otto (1957) were the first investigators to propose a useful procedure for prediction of power consumption in non-newtonian fluids using fundamental viscometric data. It was found that the fluid motion in the vicinity of the impeller could be characterized by relating shear rate to impeller speed as follow:-

$$\text{Shear rate} \propto N \quad (5.9)$$

From figure 3.14 and the above equation, it can be concluded that viscosity can be expressed a function of speed.

The data of experiments plotted in Figure 5.5 showed that a longer mixing time obtained by 19 cSt. viscosity of lubricating oil than by 12 cSt. viscosity of lubricating oil. This is caused from the higher difference in the viscosity and lower shear rate of lubricating oil.

The difference in viscosities of the components and final product provide the "driving force", the greater the differences in viscosities of the components, the more horsepower is required. Also, the higher the final product viscosity, the more power required.

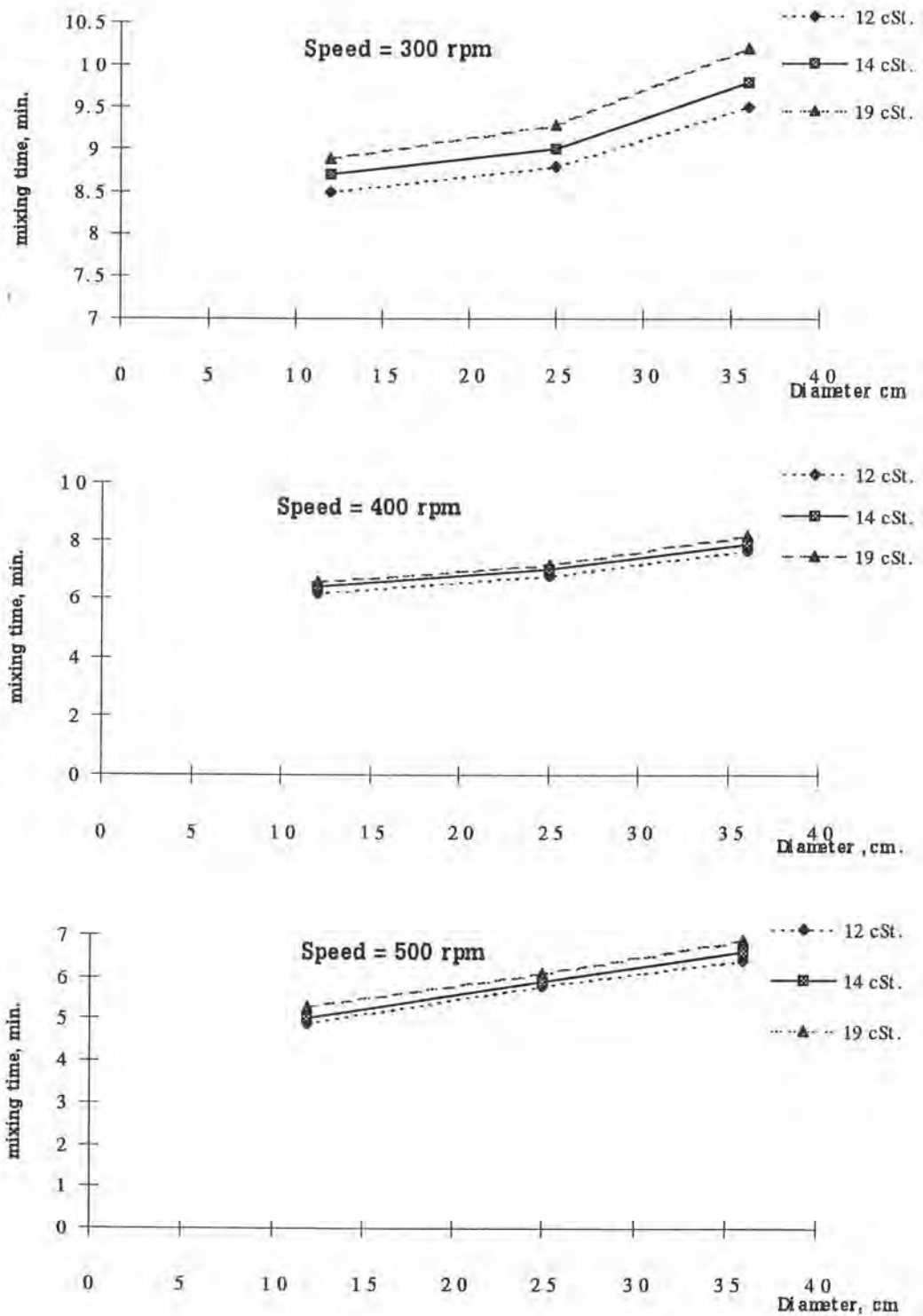


Figure 5.4 Relationship between mixing time and diameter tank

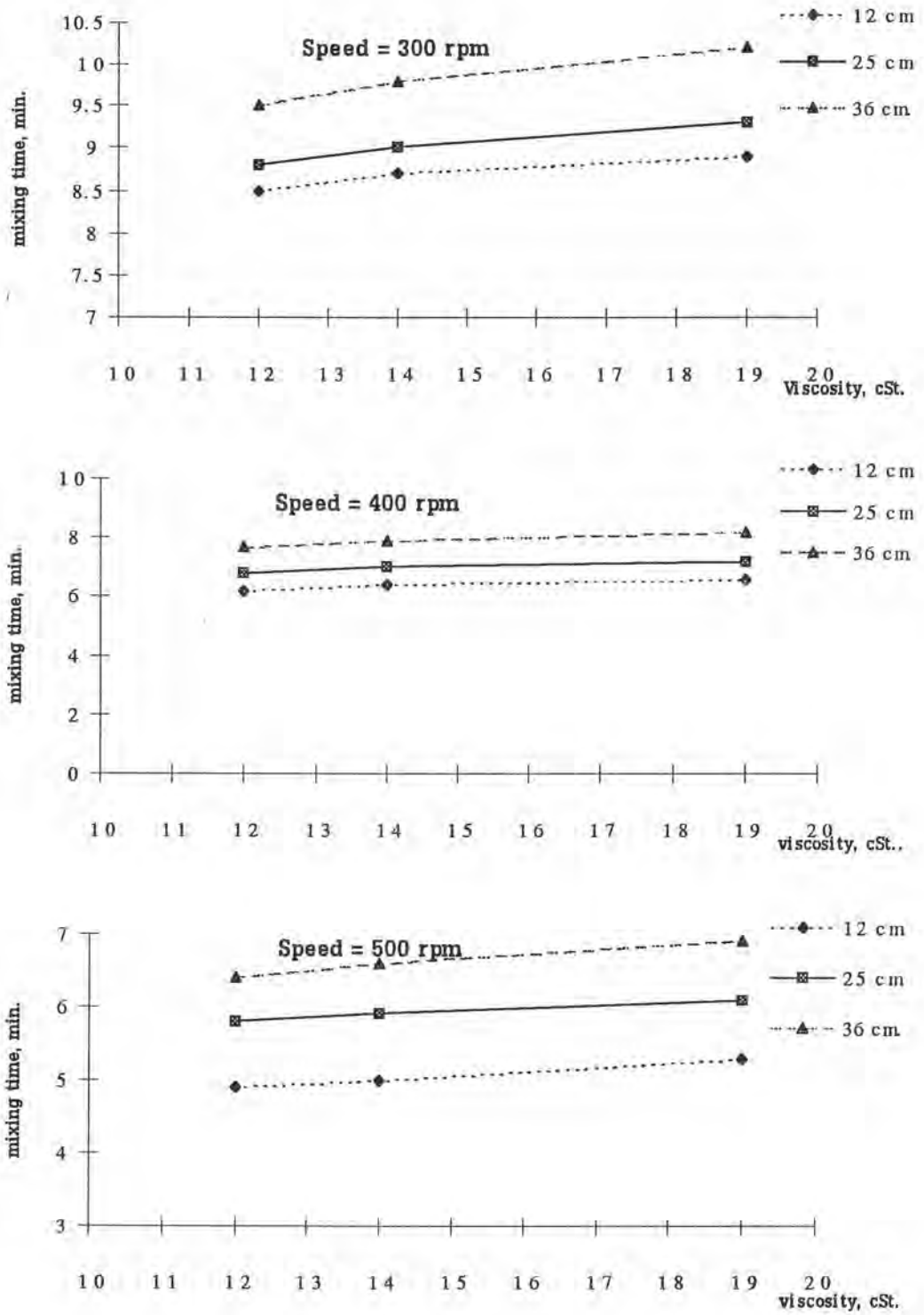


Figure 5.5 Relationship between mixing time and viscosity

5.4 Model development

Since it is neither convenient nor economical to experiment with industrial size units to get optimal process conditions, it is desirable to have a scale-up technique to predict the mixing time of industrial scale. From experiment, it can be observed that the mixing time may be related with operating conditions, such as : rotational speed, vessel geometry and any physical properties of lubricating oil. So, the many variables of any physical properties and operating conditions can be conveniently related by using suitable dimensionless parameters and combined them to arrive at two parameters that related all the variables involved in the mixing process.

For geometrically similar tanks; such as : constant impeller, tank geometric ratios, location of addition, the dimensionless mixing time should depend on :

$$Nt_m = f\{\rho N D_i^2 / \mu, \Delta\rho g H_1 / \rho N^2 D_i^2, \mu_2 / \mu_1, H_1 / D_i\} \quad (5.10)$$

Where the second dimensionless group on the right-hand side is Richardson number, R_i , and H_1 is depth of liquid in tank.

This general dependence can be simplified to :

$$\tau = Nt_m = f\{R_o, R_i\} \quad (5.11)$$

Richardson number is related to the density differences in two-component or two stratified layers. For this study, the experiments were conducted in multi-component system. So Reynolds number is a more proper dimensionless for mixing time to use.

The Reynolds number, R_o , as defined $\rho D_i^2 N / \mu$, which represents the ratio of inertial forces to viscous forces. The dimensionless of mixing time, τ , as defined Nt_m , which represents the ratio of blending time to a reference time.

Dimensionless parameters, R_0 and τ for all experiments are summarized in Table 5.6

The results in Table 5.5 can be plotted as shown in Figure 5.4. The data points fall on different curves which are difficult to explain or predict mixing time, However, when using logarithmic plot of τ against R_0 , the data points can be shown as Figure 5.5.

With the logarithmic plot of τ against R_0 , it can be observed that a straight line correlation fits the data points. A linear correlation analysis provides the following equation,

$$\tau = 0.367 R_0 + 2582.42 \quad (5.12)$$

Information on the mixing time from the xperiment and from this correlation should be compared as shown in Appendix I, showing relative errors between the mixing time from experimental and calculated mixing time from the correlation. The maximum relative error is 10.14 %.

Table 5.7 Dimensionless parameters

Viscosity of lubricating oil @ 100 °C, cSt.	Diameter of impeller, cm.	Speed, rpm.	Dimensionless		
			R_o	τ	
12	4	300	152.145	2550	
		400	202.860	2480	
		500	253.575	2450	
	8.3	300	655.079	2640	
		400	873.439	2720	
		500	1091.799	2900	
	12	12	300	1369.306	2850
			400	1825.741	3080
			500	2282.176	3200
14	4	300	140.350	2610	
		400	187.133	2560	
		500	233.917	2500	
	8.3	300	604.295	2700	
		400	805.726	2800	
		500	1007.158	2950	
	12	12	300	1263.150	2940
			400	1684.201	3160
			500	2105.250	3300
19	4	300	79.365	2670	
		400	105.821	2640	
		500	132.276	2650	
	8.3	300	341.718	2790	
		400	455.524	2880	
		500	569.929	3050	
	12	12	300	714.289	3060
			400	952.685	3280
			500	1190.482	3450

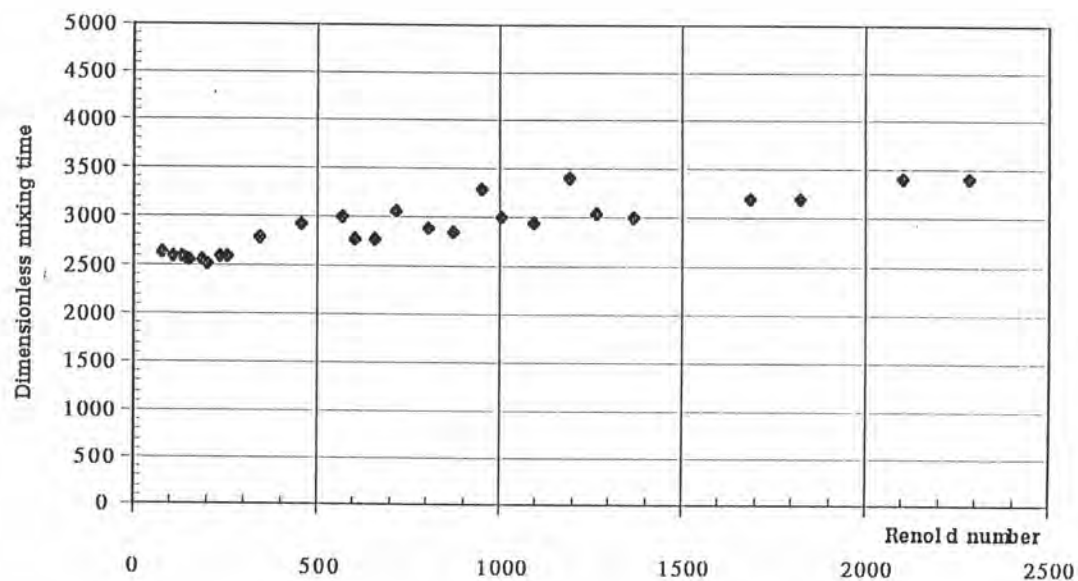


Figure 5.6 Dimensionless mixing time plotted against Reynolds number

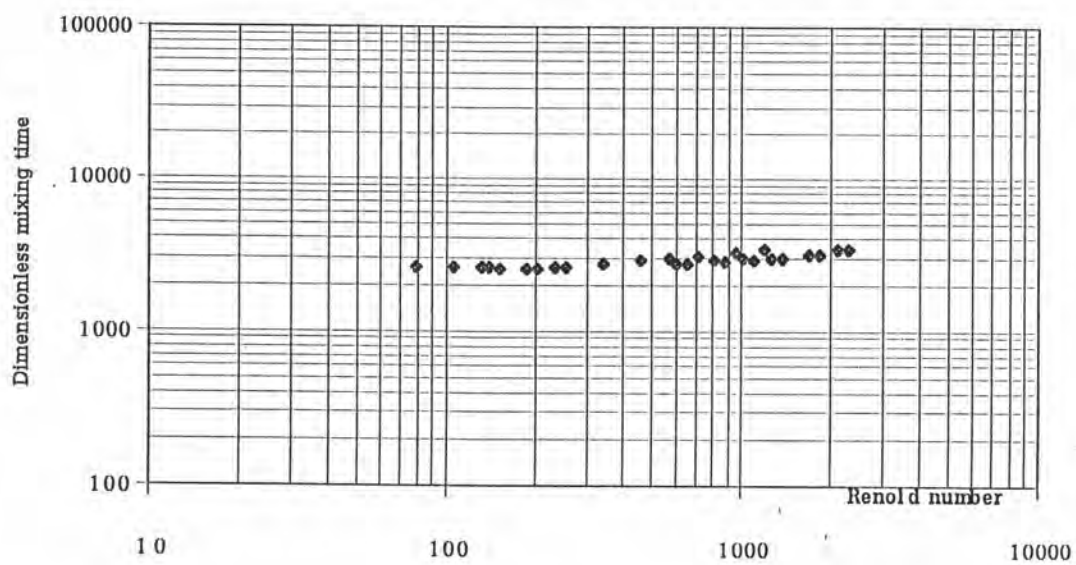


Figure 5.7 Dimensionless mixing time plotted linearly against Reynolds number

5.5 Optimization of energy requirements

The measurement of power is an important characteristic of the mixer performance for establishing operating costs. From this experiment, during the mixing process, the digital multimeter was used to measure the electrical current (AC-current). The electrical current was used to calculate both power and the total energy consumption as in Table 5.8.

Table 5.8 The total energy consumption

Viscosity of lubricating oil @ 100 Deg C, cSt.	Diameter of impeller, cm	Speed, rpm	Current, Amp.	Power, Watt.	Energy consumption, watt-sec.
12	4.0	300	0.00019	0.0376	19.1862
		400	0.00052	0.1030	38.3011
		500	0.00081	0.1604	47.1517
	8.3	300	0.00652	1.2910	681.6269
		400	0.01595	3.1581	1288.5048
		500	0.02457	4.8649	1692.9713
	12.0	300	0.03742	7.4092	4223.2212
		400	0.07139	14.1352	6530.4716
		500	0.10154	20.1049	7720.2893
14	4.0	300	0.00018	0.0356	18.6041
		400	0.00046	0.0911	34.9747
		500	0.00091	0.1802	54.0540
	8.3	300	0.00743	1.4711	794.4156
		400	0.01641	3.2492	1364.6556
		500	0.02863	5.6687	2006.7340
	12.0	300	0.03824	7.5715	4452.0538
		400	0.07396	14.6441	6941.2939
		500	0.10893	21.5681	8540.9834
19	4.0	300	0.00020	0.0396	21.1464
		400	0.00053	0.1049	41.5562
		500	0.00104	0.2059	65.4826
	8.3	300	0.00789	1.5622	871.7188
		400	0.01783	3.5303	1525.1069
		500	0.03782	7.4884	2740.7398
	12.0	300	0.05242	10.3792	6352.0459
		400	0.11296	22.3661	11004.1114
		500	0.15406	30.5039	12628.6063

It would be an interesting to investigate the power consumption in each condition. This would be useful in evaluating an appropriate mixing condition as follows:-

At 300 rpm, the shorter required mixing time implies that lower energy consumption would be obtained. On the other hand, longer required mixing time and higher energy consumption would be obtained from 500 rpm of rotational speed. So, 400 rpm of rotational speed of impeller is the best mixing condition because it can be applied to conserve energy, save time and increase the production capacity.

5.6 Analyses of the physical properties of lubricating oils

Samples were checked for other properties after completion of the mixing process to ensure that the product are within specifications as shown in Table 5.9.

From the result of physical properties, it was found that the variation of result may be caused by two main reasons. Firstly, the quantities of component had not been corrected in the formula so the final results were a little deviated value. Secondly, the limitation of sensitivity of measuring instrument must be considered. However, the acceptable variation limit of test results were obtained within specifications.

Table 5.9 Physical properties of lubricating oils

Viscosity of lubp. oils, @100 °C, cSt	Diameter tank,cm.	Speed rpm.	Physical properties					
			Density, kg/l.	Viscosity @100 °C	Pour point, °C	Crackle test	Ca, % wt.	Foam test
12	12	300	0.9027	11.85	-27	neg.	0.90	0
		400	0.9036	12.14	-30	neg.	0.94	0
		500	0.9041	11.90	-21	neg.	0.89	0
	25	300	0.9052	12.10	-21	neg.	0.87	0
		400	0.9051	12.31	-21	neg.	0.94	0
		500	0.9032	11.94	-27	neg.	0.87	0
	36	300	0.9032	11.50	-27	neg.	0.88	0
		400	0.9034	12.2	-18	neg.	0.94	0
		500	0.9036	12.00	-24	neg.	0.90	0
14	12	300	0.9167	14.1	-24	neg.	1.07	0
		400	0.9157	14.6	-27	neg.	1.06	0
		500	0.9154	14.0	-24	neg.	1.15	0
	25	300	0.9141	14.4	-24	neg.	1.13	0
		400	0.9161	14.1	-24	neg.	1.16	0
		500	0.9158	14.4	-27	neg.	1.07	0
	36	300	0.9135	14.8	-24	neg.	1.06	0
		400	0.9141	14.2	-24	neg.	1.08	0
		500	0.9156	14.3	-30	neg.	1.15	0
19	12	300	0.9350	18.7	-24	neg.	2.53	0
		400	0.9357	19.0	-24	neg.	2.36	0
		500	0.9354	18.9	-24	neg.	2.50	0
	25	300	0.9342	18.9	-21	neg.	2.58	0
		400	0.9367	19.0	-27	neg.	2.54	0
		500	0.9350	19.2	-32	neg.	2.32	0
	36	300	0.9357	20.1	-24	neg.	2.61	0
		400	0.9343	19.8	-27	neg.	2.44	0
		500	0.9333	20.0	-24	neg.	2.69	0

5.7 Optimization in industrial scale

To predict the scale-up performance of agitated vessel in production size mixing system, the performance equation (5.12) obtained from the experiment can be used to calculate the required mixing time in lubricating oils of blending plant, i.e. for 10,000 liters of in blending tank, having 233.5 cm. of tank diameter, the mixing time can be calculated from performance equation as Table 5.10.

Table 5.10 The required mixing from performance equation

Product	R_s	t_m , min
STD-Oil-1	50729.1	53
STD-Oil-2	40919.8	44
STD-Oil-3	35430.2	39

As the required mixing time of all products in blending process was 60 minutes, the samples were taken at t_m and 60 minutes for verifying that all physical properties were in controlled specifications. The result of physical properties of lubricating oil at t_m and 60 minutes are shown in Table 5.11.

From the test results, it was found that both test results were within specifications. So, this performance equation can be used to apply in lubricating oil blending plant because of time saving, energy conservation and increase the production capacity.

Table 5.11 Test result of finished lubricating oil in blending plant

Properties	STD-Oil-3		STD-Oil-2		STD-Oil-1	
	$t_m = 39$	$t_m = 60$	$t_m = 44$	$t_m = 60$	$t_m = 53$	$t_m = 60$
Density @ 15 Deg C, kg/l	0.8750	0.8730	0.8860	0.8879	0.9039	0.9039
VK. @ 100 Deg C, cSt.	19.1	19.0	14.4	14.4	11.82	11.83
Pour point, Deg C	-24	-24	-24	-24	-27	-27
Crackle test	neg.	neg.	neg.	neg.	neg.	neg.
Ca content, % Wt.	0.126	0.129	0.127	0.125	0.902	0.902
Foam test	0	0	0	0	0	0

The specification of STD-Oil-1,2,3 as shown in appendix J