

CHAPTER IV

RESULTS AND DISCUSSION

Raw Material Analysis.

Unpurified rosin or original rosin (about 40 mg) was esterified with excess diazomethane and the ester products were characterized by GC-MS. The GC-MS chromatogram in Figure A 1 was obtained using in a DB-1 capillary column, shows that the original rosin is composed of six resin acids. The percent composition and relative retention times are presented in Figure 4.1 and Table 4.1. The weight percent of the six resin acids in rosin was determined directly from the normalized areas of the peaks.

Table 4.1 Relative retention time and weight percent composition of rosin.

Original Rosin	Peak No.	Retention time	Area	%Area	Name
	1	12.25	45,966	9.64	methyl pimarate
	2	12.37	7,606	1.59	methyl sandaracopimarate
	3	12.56	79,973	16.77	methyl palustrate
	4	12.88	19,696	4.13	methyl dehydroabietate
	5	13.56	251,712	52.78	methyl abietate
	6	14.40	71,973	15.09	methyl neoabietate
Total			476,926	100.00	

The ^1H -NMR and ^{13}C -NMR spectra are shown in Figures A 2 and A 3 respectively. The spectra indicate that abietic acid is the main component of the rosin. The prominent peaks were arisen from the conjugated double bond at δ 5.60 ppm for ^1H and δ 120.35, 122.62, 135.52 and 145.10 ppm.

Mass spectra of the 6 resin acids were compared with mass spectra of standard compounds in the library program support on the GC-MS. (Figure A 4). The order arrangement of the six resin acids in relative retention time are pimaric, sandaracopimaric, palustric, dehydroabietic, abietic, and neoabietic acid respectively.

MS data of the identified resin acids and the relative abundance of their parent ions M^+ are given in Table 4.2 [26, 27]. This data was compared with relative abundance of the parent ion M^+ of starting rosin for confirming their structures. (Table 4.3)

A determined melting point of original rosin by DSC was 109.1°C as shown in Figure A 5.

Table 4.2 Relative abundances in the recorded mass spectra of methyl esters of identified resin acid.

GC-MS peak number	Relative abundances of the 5 most prominent peak (% of base peak)	Parent ion M^+ (m/e/%)
1	121/100, 180/25, 91/18, 119/14, 105/14	316/6
2	121/100, 91/16, 119/15, 133/15, 93/14	316/7
3	241/100, 105/84, 301/82, 91/79, 149/68	316/61
4	239/100, 240/21, 299/12, 141/11, 129/10	314/10
5	256/100, 121/82, 241/72, 213/71, 105/68	316/48
6	135/100, 121/32, 148/25, 134/19, 91/18	316/7

Table 4.3 Relative abundances in the recorded mass spectra of the raw material rosin

GC-MS peak number	Relative abundances of the most 5 prominent peak (% of base peak)	Parent ion M ⁺ (m/e/%)
1	121/100, 180/20, 91/22, 119/18, 105/15	316/4
2	121/100, 91/25, 119/15, 133/16, 93/20	316/7
3	241/100, 105/84, 301/75, 91/70, 149/50	316/45
4	239/100, 240/21, 299/12, 141/11, 129/8	314/10
5	256/80, 121/100, 241/73, 213/55, 105/87	316/48
6	135/100, 121/32, 148/22, 134/19, 91/23	316/13

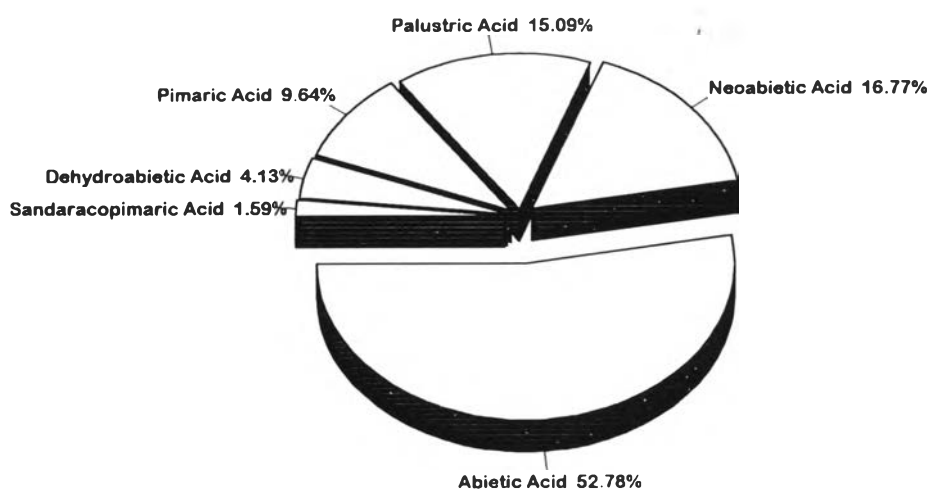


Figure 4.1 The percent by weight of resin acids in rosin

The results of experiments concerned with dehydrogenation.

The dehydrogenation reaction (Disproportionation Reaction) is a method for effectively modifying rosins to render them less susceptible to oxidation by atmospheric oxygen and for increasing the qualities of the end products. The reaction involves removal of two hydrogen atoms from the abietic-type acids and the rearrangement of the double-bond system to form an aromatic nucleus of dehydroabietic acid. The process for preparing quality rosin involves two steps: disproportionation reaction (dehydrogenation reaction) and purification.

A disproportionated rosin could be easily prepared by heating a rosin in the presence of disproportionating catalysts such as iodine, palladium on carbon, or platinum powder. The catalyst was employed preferably in an amount of 0.1 to 1.0% by weight of rosin. The preferred reaction temperature was 200 to 280°C and the optimum heating period was 3 to 5 hours. [17]

In this work the reaction was carried out at various temperatures (240, 260, and 280 °C) and under a nitrogen atmosphere in the presence of 0.3% Pd/C catalyst for 4 hours. Disproportionated rosin was distilled under reduced pressure (3 mmHg) in an atmosphere of nitrogen to give purified disproportionated rosin as the main fraction. The results are shown in Table 4.4.

After purified by distillation, dehydrogenated rosin was analyzed by NMR. The ^1H -NMR spectrum (Figure A 6) displayed the signals of aromatic proton at region δ 7.10 ppm and impurities at region δ 5.0–5.5 ppm. The ^{13}C -NMR spectrum (Figure A 7) displayed the carbonyl carbon at δ 185.81 ppm, the aromatic carbons and carbon of impurities showed at region δ 123.90–146.74 ppm.

Table 4.4 Main fractions of disproportionated rosin determined by distilling under a reduce pressure of 3 mmHg

Condition	Distilling Temp.(°C)	Yield (%)
240°C	<195	4.12
	195-250	88.44
	>250	7.44
260°C	<195	5.26
	195-250	86.35
	>250	8.39
280°C	<195	6.62
	195-250	86.41
	>250	6.97

Disproportionated rosin was esterified with excess diazomethane and was analyzed by GC-MS. Chromatograms of dehydrogenated rosins at various temperatures are shown in Figure A 8. The percentage yield and the relative retention times of dehydrogenated rosins are presented in Table 4.5 and Figure 4.2. The mass spectra of individual compound were compared with mass spectra of standard compounds from the library program as shown in Figure A 9.

The mass spectrum of dehydrogenated rosin (dehydroabiatic acid is the main component) displayed the relative abundance of percent ion at m/e : 314 (6%), 239(100%), 240(20%), 299(12%), 141(9%), 129(5%).

Purification was intended to mean the removal of high molecular weight peroxide materials which were contained in the disproportionated rosin and unsaponifiable materials which were originally contained in the disproportionated rosin. The purification could be carried out according to usual purification methods such as distillation, crystallization, and extraction.

Table 4.5 Relative retention times and percentage yields of dehydrogenated rosin at various temperatures

Condition	Peak No.	Retention Time	Area	%Area	Name
240°C	1	12.04	12,395	1.84	methyl dihydropimarate
	2	12.93	661,060	98.16	methyl dehydroabietate
Total			673,455	100.00	
260°C	1	12.06	3,540	3.75	methyl dihydropimarate
	2	12.84	1,520	1.61	methyl dihydroabietate
	3	12.93	89,257	94.64	methyl dehydroabietate
Total			94,317	100.00	
280°C	1	12.04	16,672	2.07	methyl dihydropimarate
	2	12.82	11,571	1.44	methyl dihydroabietate
	3	12.95	776,836	96.49	methyl dehydroabietate
Total			805,079	100.00	

In this research, the purification was carried out by distillation and crystallization. Dehydrogenated rosin obtained by this method was recovered about 85% yield of disproportionated rosin (at 240°C =86.13%, 260°C=84.37% and 280°C=84.11%).

After crystallized by solvent, dehydrogenated rosin was analyzed by NMR. The ^1H -NMR spectrum (Figure A 10) displayed the signals of aromatic proton at region δ 7.10 ppm. The ^{13}C -NMR spectrum (Figure A 11) displayed the carbonyl carbon at δ 185.81 ppm and the aromatic carbons showed at δ 123.90, 123.98, 124.11, 126.92, 134.67 and 146.74 ppm. The result from ^1H -NMR and ^{13}C -NMR indicate that after crystallized the disproportionated rosin, impurities such as peroxides or dihydroabietic acids were removed to give high purity dehydroabietic acid without hydrogenation in final step. The melting point of disproportionated rosin determined by DSC was 160°C. (Figure A 12)

Disproportionated rosin was contaminated with a little dihydroabietic acids because during dehydrogenation, the two hydrogen atoms removed from abietic-type acids were readily absorbed by other two double-bond abietic acids present in the rosin to produce the stable dihydroabietic acid.

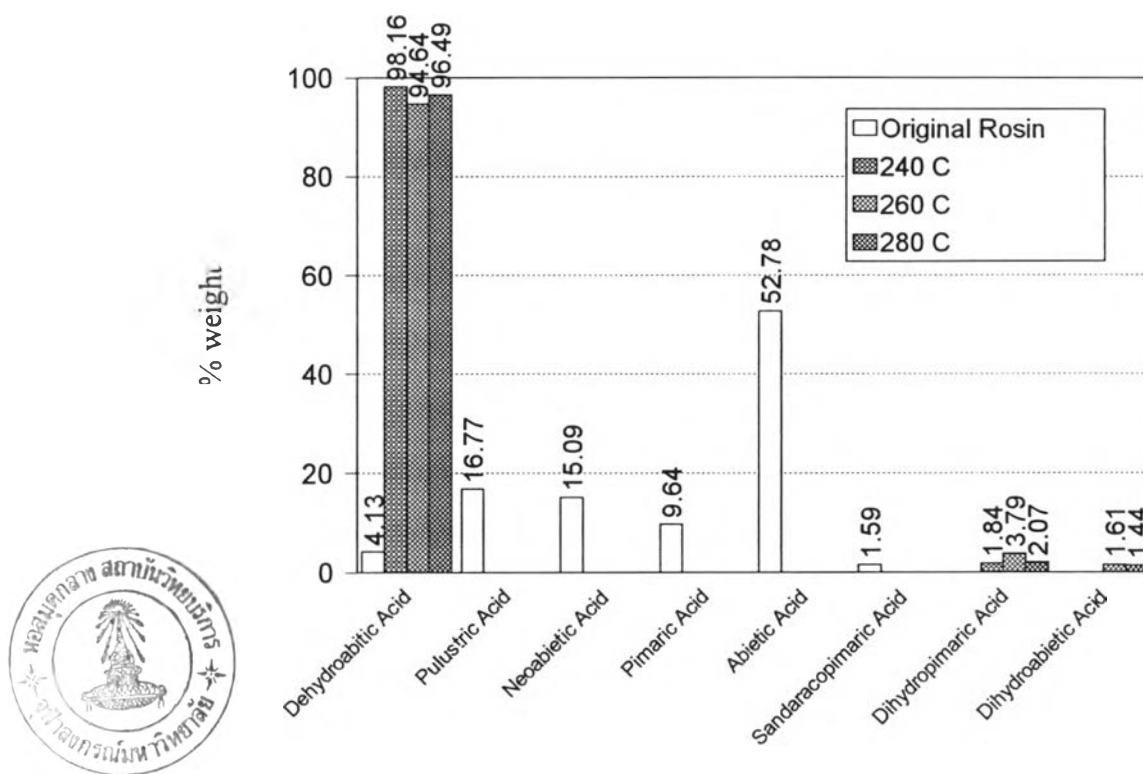


Figure 4.2 Percentage components of dehydrogenated rosin (Dehydroabietic Acid) at various temperatures

The results of experiments concerned with hydrogenation.

Hydrogenation is the most satisfactory method for decreasing the susceptibility of rosin to air oxidation and to improve quality of the end products. In this section various hydrogenation parameters were studied: catalyst type, reaction temperature, reaction time, hydrogen pressure, and catalyst concentration, to determine the optimum reaction conditions.

1. The effect of catalyst types on the hydrogenation.

In the first process, rosin was hydrogenated by using various types of catalysts such as Raney Nickel, Pt/C, Pt/Al₂O₃, Pd/C. These suitable hydrogenation catalysts were selected for treatment of rosin under fixed condition at 200°C, hydrogen pressure 500 psi, reaction time for 6 hours, catalyst concentration Raney Nickel 4%, Pt/Al₂O₃ 4%, Pd/C 1%, and Pd/C 1% by weight of rosin were used respectively. The reaction was performed in ethanol (300 ml) with 300 rpm agitation speed, in a stirred reactor as indicated in the experiment 3.1.

Hydrogenated rosins were esterified with excess diazomethane and were analyzed by GC–MS. Chromatograms of hydrogenated rosins under the various catalyst types are displayed in Figure A 13. The percentage yields and the relative retention times of the hydrogenated rosins are presented in Table 4.6 and Figure 4.3. The mass spectra of individual components were compared with mass spectra of standard compound from the library program as shown in Figure A 14.

Table 4.6 The percentage yields and relative retention times of hydrogenated rosin produced with various catalyst types

Catalysts	Peak No.	Retention Time	Area	%Area	Name
Pt/Al ₂ O ₃	1	12.02	13,891	3.20	methyl dihydropimarate
	2	12.49	7,553	1.74	methyl dihydroabietate
	3	12.58	11,364	2.62	methyl dihydroabietate
	4	12.80	187,313	43.16	methyl dihydroabietate
	5	12.88	196,186	45.20	methyl dehydroabietate
	6	13.56	17,733	4.09	methyl abietate
Total			434,040	100.00	

Table 4.6 (continued)

Catalysts	Peak No	Retention Time	Area	%Area	Name
Raney Ni	1	12.23	28,825	3.29	methyl pimarate
	2	12.51	10,317	1.18	methyl tetrahydropimarate
	3	12.56	14,319	1.63	methyl dihydroabietate
	4	12.77	495,148	56.49	methyl dihydroabietate
	5	12.91	161,929	18.47	methyl dehydroabietate
	6	13.01	130,758	14.92	methyl tetrahydroabietate
	7	13.49	35,291	4.03	ethyl dihydropimarate
Total			876,587	100.00	
Pt/C	1	11.93	4,379	1.54	methyl dihydropimarate
	2	12.25	10,708	3.76	methyl pimarate
	3	12.60	12,686	4.45	methyl dihydroabietate
	4	12.78	53,264	18.70	methyl dihydroabietate
	5	12.91	31,715	11.14	methyl dehydroabietate
	6	13.58	172,048	60.41	methyl abietate
Total			284,800	100.00	
Pd/C	1	12.04	8,762	2.41	methyl dihydropimarate
	2	12.52	34,280	9.42	methyl tetrahydropimarate
	3	12.84	238,668	65.56	methyl dihydroabietate
	4	12.93	27,582	7.58	methyl dehydroabietate
	5	13.06	15,775	4.33	methyl tetrahydroabietate
	6	13.19	7,587	2.08	ethyl tetrahydropimarate
	7	13.53	31,372	8.62	ethyl dihydroabietate
Total			364,026	100.00	

The results from these experiments indicated that Raney Nickel and Pd/C were the most suitable catalysts for hydrogenation of rosin. The results in Table 4.6 indicate that Raney Nickel catalyst is the best catalyst for production of tetrahydroabietic acid. Moreover, Raney Nickel is a simple catalyst and also available commercially that would serve the optimum economics for production of tetrahydroabietic acid. While other catalysts such as Pt or Pd gave a partial

effect for hydrogenation of rosin, these catalysts are more expensive than Raney Nickel. For this reason Raney Nickel was selected in the next steps.

2. The effect of reaction temperature on hydrogenation.

The first parameter to be varied was reaction temperature. The hydrogenation was carried out under hydrogen pressure of 500 psi, a reaction time of 8 hours, ethanol (300 ml), and concentration of 8% by weight of rosin. The temperature was varied from room temperature, 100, 150 and 200°C.

The products from these reactions were esterified with excess diazomethane and analyzed by GC–MS. Chromatograms of hydrogenated rosin are shown in Figure A 15. The percentage yields and relative retention times are presented in Table 4.7 and Figure 4.3.

Table 4.7 The percentage yield and relative retention times of hydrogenated rosin produced at various reaction temperatures

Temperatures	Peak No.	Retention Times	Area	%Area	Name
Room Temp.	1	12.25	9,400	2.62	methyl pimarate
	2	12.58	33,019	9.19	methyl palustrate
	3	12.88	25,703	7.16	methyl dehydroabietate
	4	13.56	276,192	76.90	methyl abietate
	5	14.42	14,830	4.13	methyl neoabietate
Total			359,144	100.00	
100°C	1	12.23	10,202	3.33	methyl pimarate
	2	12.50	3,270	1.07	methyl dihydroabietate
	3	12.56	21,535	7.03	methyl palustrate
	4	12.76	115,876	37.83	methyl dihydroabietate
	5	12.89	68,105	22.24	methyl dehydroabietate
	6	13.54	87,281	28.50	methyl abietate
Total			306,269	100.00	

Table 4.7 (continued)

Temperature	Peak No	Retention Times	Area	%Area	Name
150°C	1	12.23	18,477	4.48	methyl pimarate
	2	12.49	4,940	1.20	methyl dihydroabietate
	3	12.58	12,001	2.91	methyl palustrate
	4	12.75	259,911	63.06	methyl dihydroabietate
	5	12.91	82,477	20.01	methyl dehydroabietate
	6	13.01	34,376	8.34	methyl tetrahydroabietate
			412,182	100.00	
200°C	1	12.23	28,825	3.29	methyl pimarate
	2	12.51	10,317	1.18	methyl dihydroabietate
	3	12.56	14,319	1.63	methyl palustrate
	4	12.77	495,148	56.49	methyl dihydroabietate
	5	12.91	161,929	18.47	methyl dehydroabietate
	6	13.01	130,758	14.92	methyl tetrahydroabietate
	7	13.49	35,291	4.03	ethyl dihydropimarate
Total			876,587	100.00	

The mass spectra of the individual components were compared to mass spectra of standard compounds in the library program for GC–MS as displayed in Figure A 16.

The results show that at temperatures of 150 to 200°C, rosin was partially hydrogenated to produce saturated resin acids. During hydrogenation at the higher temperature, the rosin may also undergo decarboxylation and isomerization reactions in competition with hydrogenation. The decarboxylation leads to fume when it was heated. This result indicated that rosin isomers were generated by isomerization of rosin at high temperature.

According to the results in Table 4.7 the percent of hydrogenation at 200°C was much higher than that at 150°C. This result demonstrated that the optimum reaction temperature for hydrogenation of rosin 200°C.

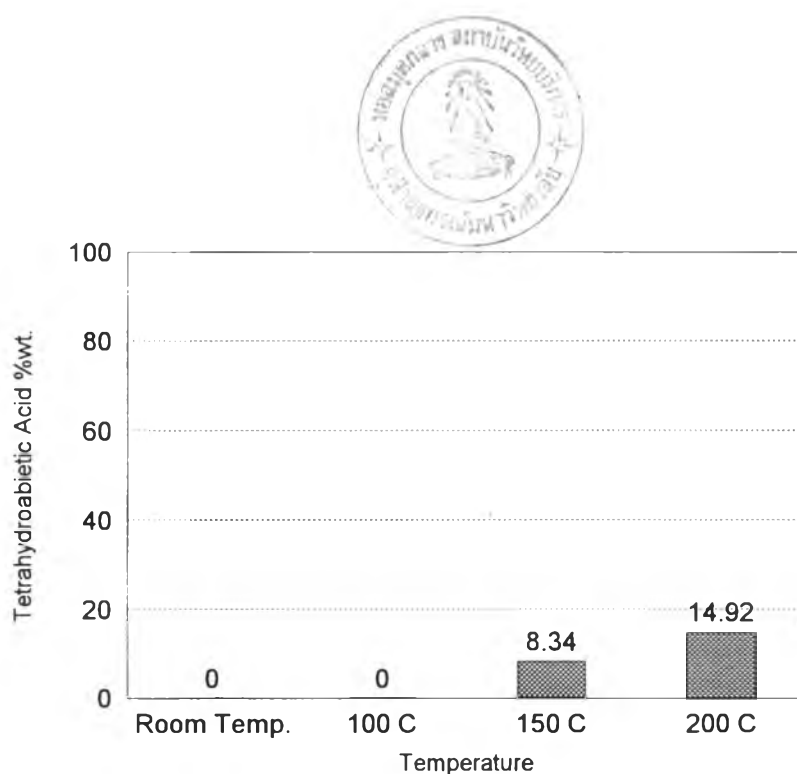


Figure 4.3 Percentage yields of hydrogenated rosin at various reaction temperatures

3. The effect of reaction time on hydrogenation.

In this part, optimum reaction time was determined by operating the hydrogenation reaction for various periods of time of 8, 10, 12, and 16 hours respectively. The reactions were performed at the optimum temperature (200°C). After a reaction was operated for the specified period of time, the reaction products were esterified with excess diazomethane and the ester products were analyzed by GC–MS. The GC–MS chromatograms of hydrogenated rosin produced after various reaction times are presented in Figure A 17.

The percentage yields of each resin acid and relative retention times are displayed in Table 4.8. Percentage yields of tetrahydroabiatic acid are shown in Figure 4.4. The mass spectra of each peak were compared to mass spectra of standard compounds in the library program for GC–MS, as shown in Figure A 18.

Table 4.8 The percentage yield and relative retention time of hydrogenated rosin produced at various reaction times

Reaction Time (hrs.)	Peak No.	Retention Time (min.)	Area	%Area	Name
8	1	12.53	8,945	1.79	methyl dihydroabietate
	2	12.73	18,083	3.61	methyl dihydroabietate
	3	12.79	94,484	18.89	methyl dihydroabietate
	4	12.94	149,061	29.79	methyl dehydroabietate
	5	13.03	188,973	37.77	methyl tetrahydroabietate
	6	13.52	29,984	5.99	ethyl dihydroabietate
	7	13.83	10,759	2.15	ethyl tetrahydroabietate
Total			500,289	100.00	
10	1	12.56	2,655	2.89	methyl dihydroabietate
	2	12.82	24,206	26.33	methyl dihydroabietate
	3	12.93	13,110	14.26	methyl dehydroabietate
	4	13.06	41,808	45.48	methyl tetrahydroabietate
	5	13.56	7,829	8.52	methyl palustrate
	6	13.86	2,328	2.53	ethyl tetrahydroabietate
Total			91,933	100.00	
12	1	12.04	4,374	1.62	methyl pimarate
	2	12.58	5,758	2.13	methyl dihydroabietate
	3	12.80	50,935	18.86	methyl dihydroabietate
	4	12.90	30,225	11.19	methyl dehydroabietate
	5	13.01	162,362	60.11	methyl tetrahydroabietate
	6	13.53	11,637	4.31	ethyl dihydroabietate
	7	13.84	4,817	1.78	ethyl tetrahydroabietate
Total			270,108	100.00	
16	1	12.58	2,845	2.14	methyl dihydroabietate
	2	12.80	25,082	18.88	methyl dihydroabietate
	3	12.90	21,388	16.10	methyl dehydroabietate
	4	13.01	64,784	48.75	methyl tetrahydroabietate
	5	13.53	13,673	10.29	ethyl dihydroabietate
	6	13.83	5,106	3.84	ethyl tetrahydroabietate
			132,878	100.00	

These experiments indicate that when the reactions were operated for a longer period of time, decarboxylation and isomerization reactions were

observed. These reactions lead to loss of product yield and make the rosin resist hydrogenation.

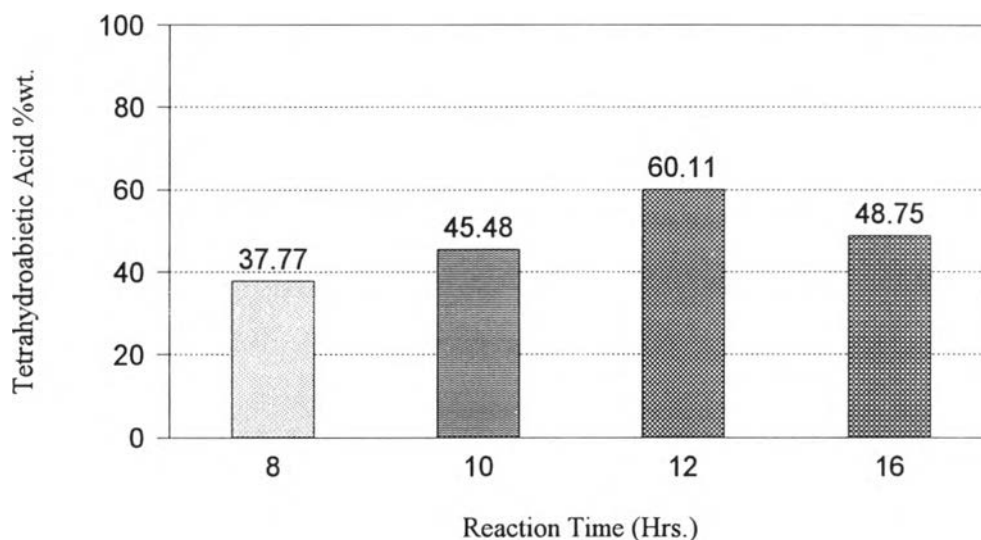


Figure 4.4 Percentage yields of hydrogenated rosin at various reaction times

The results (Figure 4.4) indicate that the percentage yield of product was increased when the reaction was performed for a longer period of time up to 12 hours. Therefore, these observations suggest that the optimum time for hydrogenation of rosin was 12 hours. This results also indicate that when the reactions are operated for longer time than optimum condition, a lower percentage yield of hydrogenated products will result. These result suggest that if the reaction is operated for a longer period of time than optimum, other reactions such as decarboxylation and isomerization compete, the percentage yield of hydrogenated rosin.

4. The effect of hydrogen pressure on hydrogenation.

Previously, reaction temperature and reaction time were considered while the hydrogen pressure was constant at 500 psi. Thus the hydrogen pressure was the next parameter of interest. The effect of hydrogen pressure on hydrogenation was studied by varying from 500, 600 and 700 psi under constant optimum parameters: reaction temperature 200°C, reaction time 12 hours, 8% catalyst concentration, and ethanol (300 ml).

The products from reactions operated at the various hydrogen pressures were esterified with excess diazomethane and the ester products were analyzed by GC–MS. Chromatograms of hydrogenated rosin esters are shown in Figure A 19. The percentage yields of individual peaks and relative retention times are presented in Table 4.9 and Figure 4.5. The mass spectra of individual compounds were compared with mass spectra of standard compounds in the library program for GC–MS as shown in Figure A 20.

Table 4.9 The percent yields and relative retention times of hydrogenated rosin produced at various hydrogen pressures

H ₂ Pressure	Peak No.	Retention Time	Area	%Area	Name
500 psi	1	12.58	5,772	2.17	methyl dihydroabietate
	2	12.80	50,958	19.13	methyl dihydroabietate
	3	12.90	30,225	11.35	methyl dehydroabietate
	4	13.01	162,518	61.01	methyl tetrahydroabietate
	5	13.53	11,824	4.44	ethyl dihydroabietate
	6	13.84	5,092	1.91	ethyl tetrahydroabietate
Total			266,389	100.00	

Table 4.9 (continued)

H ₂ Pressure	Peak No.	Retention Time	Area	%Area	Name
600 psi	1	12.60	6,793	2.16	methyl dihydroabietate
	2	12.82	72,302	23.03	methyl dihydroabietate
	3	12.93	26,002	8.28	methyl dehydroabietate
	4	13.06	202,651	64.57	methyl tetrahydroabietate
	5	13.86	6,165	1.96	ethyl tetrahydroabietate
Total			313,913	100.00	
700 psi	1	12.06	4,417	1.12	methyl dihydropimarate
	2	12.60	5,923	1.50	methyl dihydroabietate
	3	12.84	63,162	15.97	methyl dihydroabietate
	4	12.95	21,406	5.41	methyl dehydroabietate
	5	13.06	286,772	72.51	methyl tetrahydroabietate
	6	13.49	5,601	1.42	ethyl dihydropimarate
	7	13.86	8,216	2.08	ethyl tetrahydroabietate
Total			395,497	100.00	

The results from GC-MS illustrate that the reaction products were increase, when the reactions were operated under higher hydrogen pressure. These reaction were observed under hydrogen pressure from 500 to 700 psi. Reaction under higher pressure than 700 psi could not be investigated because the hydrogen gas regulator was limited at 700 psi.

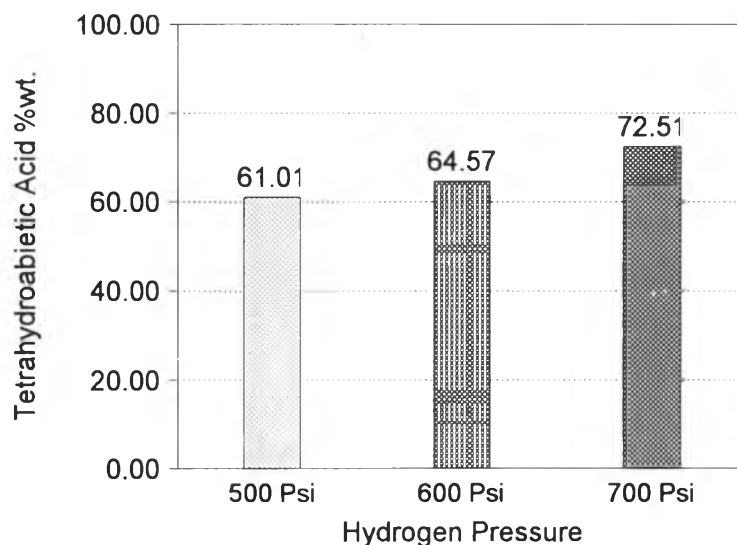


Figure 4.5 Percent yields hydrogenated rosin at various hydrogen pressure

Therefore, with the current apparatus the optimum hydrogen pressure was 700 Psi.

5. The effect of catalyst concentration on hydrogenation.

The last variable parameter to be considered for the hydrogenation reaction was catalyst concentration. The Hydrogenation reaction was performed under optimum parameters (200°C, 12 hours, and 700 psi hydrogen) with varying catalyst concentrations of 8, 10 and 12% wt of rosin. The products from this reaction were esterified with excess diazomethane and the ester products were analyzed by GC-MS. Chromatogram of hydrogenated rosins are shown in Figure A 21. Percentage yields and relative retention time are presented in Table 4.10. The percentage hydrogenation of rosin is plotted against catalyst concentration in the reaction as shown in Figure 4.6. The mass spectra of each compound was compared with mass spectra of standard compounds in the library program as shown in Figure A 22.

Table 4.10 The percentage yields and relative retention times of hydrogenated rosin produced at various catalyst concentration

% Catalyst by wt of rosin	Peak No.	Retention Time	Area	% Area	Name
8	1	12.06	4,417	1.12	methyl pimarate
	2	12.60	5,923	1.50	methyl dihydroabietate
	3	12.84	63,162	15.99	methyl dihydroabietate
	4	12.95	21,406	5.42	methyl dehydroabietate
	5	13.08	286,772	72.60	methyl tetrahydroabietate
	6	13.49	5,085	1.29	ethyl dihydropimarate
	7	13.86	8,216	2.08	ethyl tetrahydroabietate
Total			394,981	100.00	
10	1	12.60	6,793	1.86	methyl dihydroabietate
	2	12.82	40,063	10.96	methyl dihydroabietate
	3	12.93	13,495	3.69	methyl dehydroabietate
	4	13.06	289,782	81.77	methyl tetrahydroabietate
	5	13.86	6,271	1.72	ethyl tetrahydroabietate
Total			365,404	100.00	
12	1	12.58	7,525	1.98	methyl dihydroabietate
	2	12.80	60,477	15.91	methyl dihydroabietate
	3	12.91	27,540	7.24	methyl dehydroabietate
	4	13.01	277,760	73.06	methyl tetrahydroabietate
	5	13.84	6,904	1.82	ethyl tetrahydroabietate
Total			380,206	100.00	

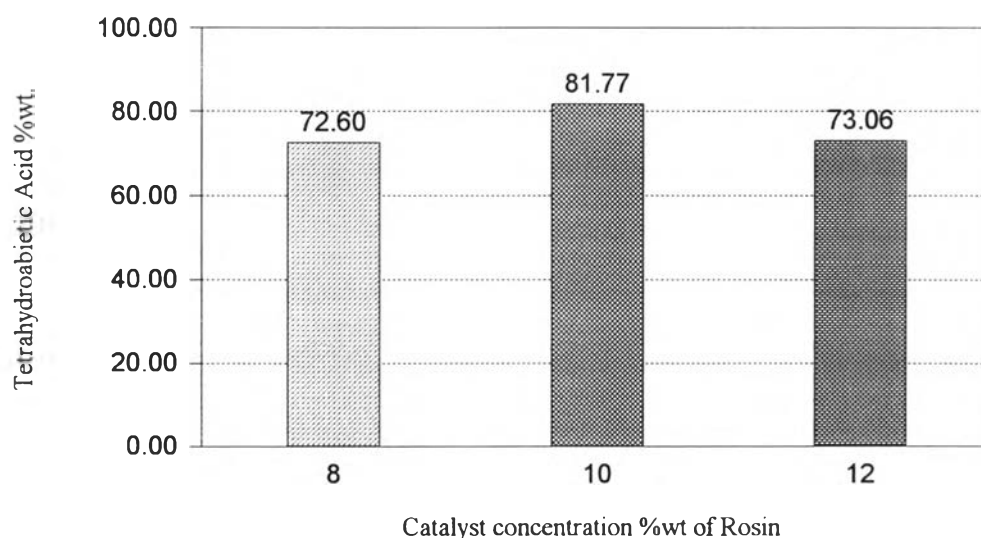


Figure 4.6 Percentage yields hydrogenated rosin at various catalyst concentrations

The results from this experiment (Figure 4.6) show that when the catalyst concentration is increased, increased yield of tetrahydroabietic acid is obtained initially. When the catalyst concentration was raised above 10% wt of rosin, yield decreased. This result suggests that the optimum catalyst concentration is about 10% by weight of rosin..

In conclusion, the results from this research illustrate that the optimum conditions for hydrogenation of rosin using Raney Nickel catalyst are 200°C, 12 hours, 700 psi hydrogen, and 10% catalyst by weight of rosin, respectively.

The rosin was hydrogenated at the above optimum conditions in the presence of Raney Nickel 10% by weight of rosin. After the reaction took place, the hydrogenated product was distilled under reduced pressure (3 mmHg) in an atmosphere of nitrogen. Purified hydrogenated rosin was observed as the main fraction in 86.23% yield. The melting point of

hydrogenated rosin was 81.3°C as determined by DSC as shown in Figure A 23.

The ^1H -NMR spectrum (Figure A 24) displays the signals of an aromatic at δ 6.9–7.2 ppm. The ^{13}C -NMR spectrum (Figure A 25) displays the carbonyl carbon at δ 186.02 ppm. For hydrogenated rosin from this experiment, signals of aromatic carbon and unsaturated carbon are displayed from δ 124–147 ppm, but the intensities are small when compared with the signal of the carbonyl carbon. The spectrum suggests that a few aromatic isomers remains from the original rosin and some new aromatic molecules might be generated from the dehydrogenation reaction.

For the batch process of US. Pat. No. 4,507,228 reported that the hydrogenation was performed under hydrogen pressure between 1500–4500 psi resulting in 75–90% yield of saturated resin acids. This research found that when the reaction was done in the optimum condition in the presence of Raney Nickel catalyst, under hydrogen pressure only 700 psi, it gave tetrahydroabietic acid (86.23% yield) almost equivalent to the reported yield in the previous patent. Therefore, the hydrogenation reaction system for rosin from our research has more advantage than the reported previously [14].