CHAPTER IV

RESULTS AND DISCUSSION

The main propose of this study was to determine in a systematic manner suitable conditions for obtaining a high yield of fuel from HDPE wax. A hydroisomerization process with a bifunctional catalyst was used. The Pt-F/Al_2O_3 catalyst was selected because platinum is the only metal that has activity for the desired reaction without undesired reactions and fluorine acts as a promoter by increasing the activity of the acidic function. Alumina is the support of choice because it is inexpensive and easy to aggregate with platinum to maintain a high metal dispersion. The catalyst prepared in this manner contained 0.3% of platinum and 0.5% of fluorine.

4.1 Characterization of platinum fluorided on alumina catalyst

The plot of x-ray diffraction data of platinum fluorided alumina catalyst is shown in Figure A2. It is similar to a plot of alumina (Figure A1). X-ray diffraction data of these only show high intensity peaks characteristic of alumina. The catalyst from this study showed high density peaks characteristic of alumina, but the catalyst from US. Pat. No. 4,923,841 [41] showed high intensity peaks characteristic of aluminium fluoride hydroxide hydrate as well as the peaks normally associated with gamma alumina (Figure A3).

A plot of x-ray diffraction data of commercial catalyst is shown in Figure A4. It show high density peaks characteristic of alumina similar to the plot of x-ray diffraction of the catalyst from this study.

Plots of scanning electron microscope / electron dispersive x-ray data of alumina and platinum fluorided on alumina catalyst are shown in figures A5 and A6 respectively. Alumina showed high intensity peaks characteristic of aluminium (Al) and oxygen (O). The catalyst from this study is shown high intensity peak characteristic of aluminium (Al), oxygen (O), platinum (Pt) and fluorine (F).

4.2 Hydroisomerization process

In this research, the hydroisomerization reactions were studied as a function of processing variables, i.e. catalyst concentration, temperature, hydrogen pressure and reaction time. Products were identified by GC/MS. Quantitative analysis was performed by gas chromatography. The change in product as a function of the above variables allowed for the choice of suitable conditions.

4.2.1 Effect of catalyst concentration

The result of GC/MS, GC analysis of the composition of products from hydroisomerization of HDPE wax as a function of catalyst concentration, keeping other processing conditions constant, are shown in Figures B1, B6, B7 and Table B1, B2, B9, B10, respectively. HDPE wax conversion and the composition trend as a function of above processing variables are shown in Table 4.1 and Figure 4.1, 4.2, respectively.

It can be seen that products consist of C_{12} - C_{25} hydrocarbons and C_{13} - C_{16} hydrocarbons are found to be the main components. The MWD was unchanged when the amount of catalyst was varied. As seen in Table 4.1, the catalyst concentration range 5-9 % by wt is indicated as satisfactory, but economic considerations dictate the use of minimum catalyst consistent with high conversion. Hence, a catalyst concentration in the vicinity of 5 % by wt could be considered optimal.

Table 4.1 HDPE wax conversion from hydroisomerization as a function of catalyst concentration

Catalyst Concentration	% conversion	
(% by wt)	first time	second time
3	-	-
4	-	-
5	100	100
6	100	100
7	100	100
8	100	100
9	100	100

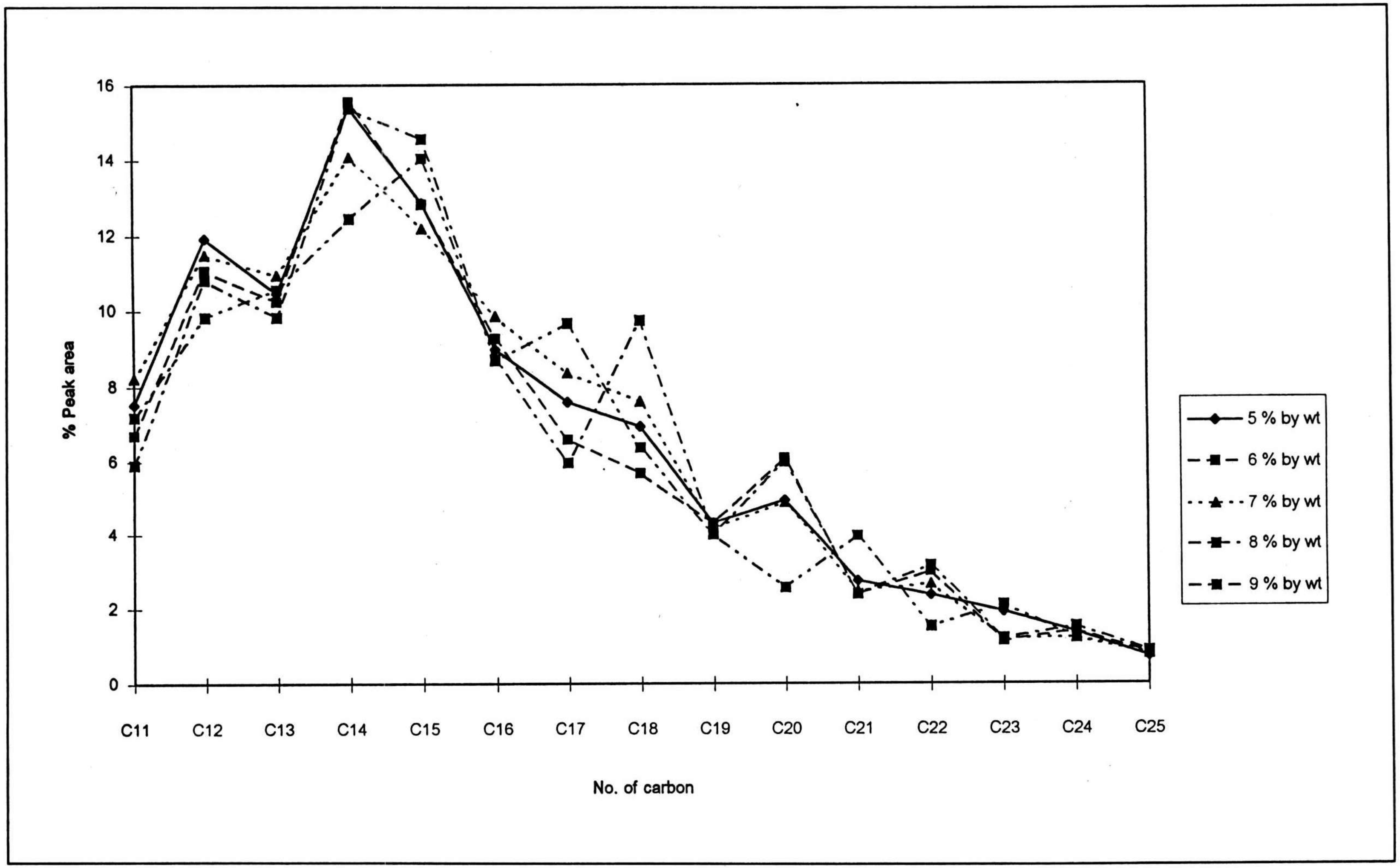

Figure 4.1 Composition trend of product from hydroisomerization as a function of catalyst concentration

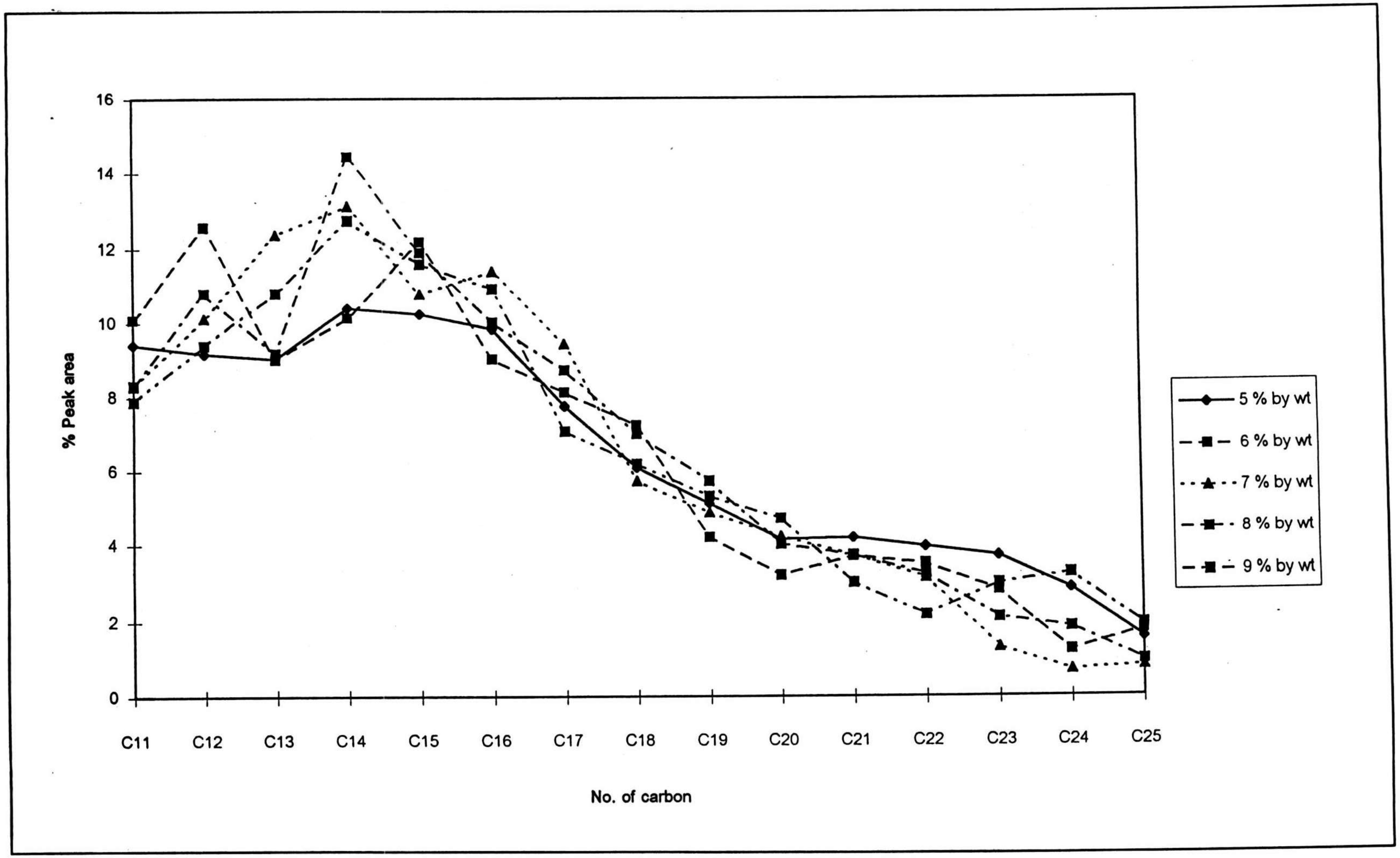

Figure 4.2 Composition trend of product from hydroisomerization as a function of catalyst concentration (second time)

4.2.2 Effect of temperature

GC/MS, GC chromatogram and composition of products from hydroisomerization as a function of temperature, using an otherwise constant set of processing conditions, are shown in Figures B2, B8 and Tables B3, B4, B11, B12, respectively. HDPE wax conversion and the composition trend as a function of above processing condition are shown in Table 4.2 and Figure 4.3, 4.4, respectively.

The result shows that the products are C_{11} - C_{25} hydrocarbons with C_{12} - C_{16} being the main components. The main trend observed is that an increase in temperature has a beneficial effect in increasing the yield of lower molecular weight components (C_{11} - C_{14}), while decreasing in the yield of higher molecular weight component (C_{23} - C_{25}). However, due to the economic considerations and the gradual change of MWD of the product when compared with the increasing of the reaction temperature, the reaction temperature of 300 °C could be considered optimal.

Table 4.2 HDPE wax conversion from hydroisomerization as a function of reaction temperature

Temperature	% conversion	
(°C)	first time	second time
150	-	-
250	-	-
300	100	100
350	100	100
400	100	100

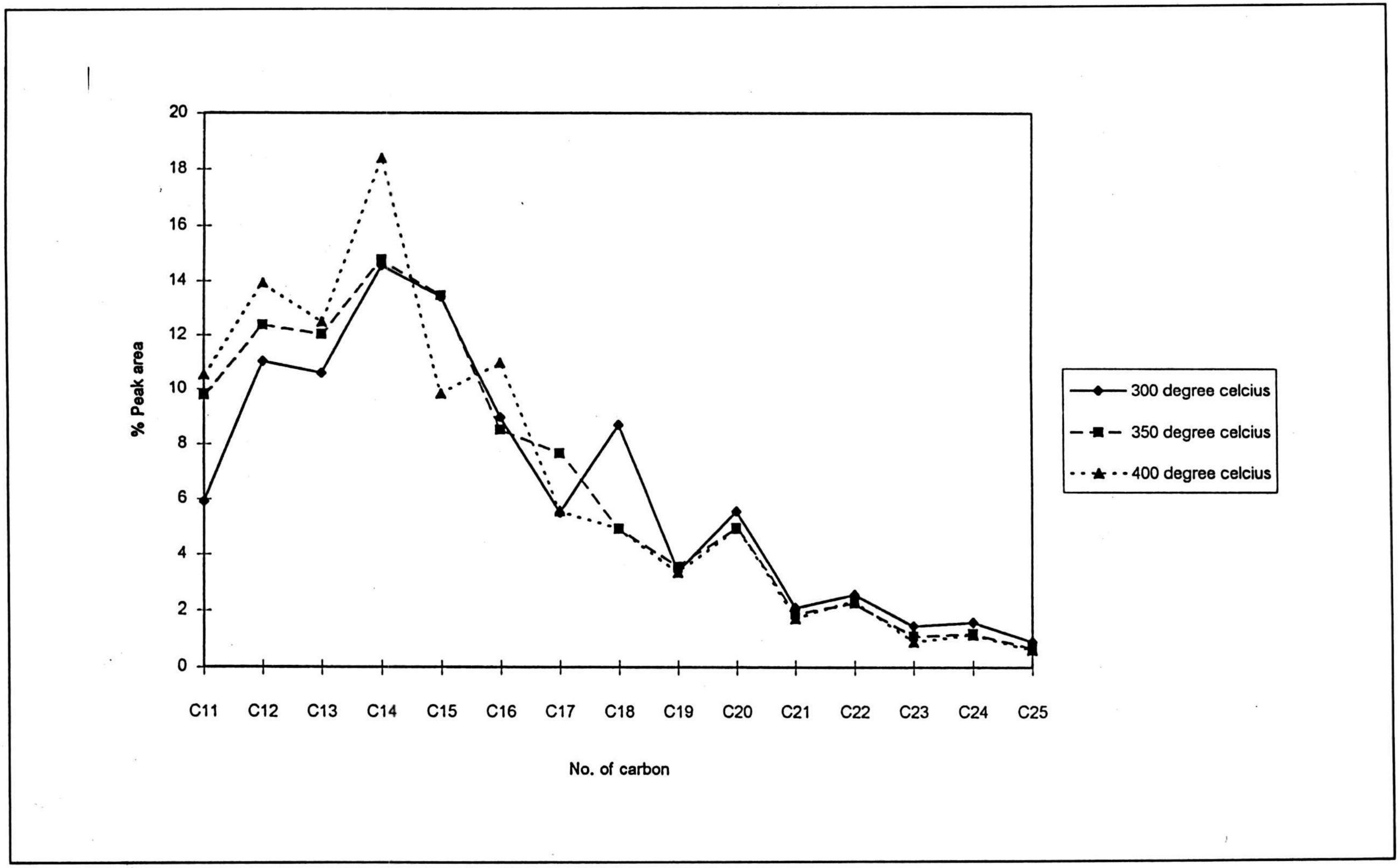

Figure 4.3 Composition trend of product from hydroisomerization as a function of temperature

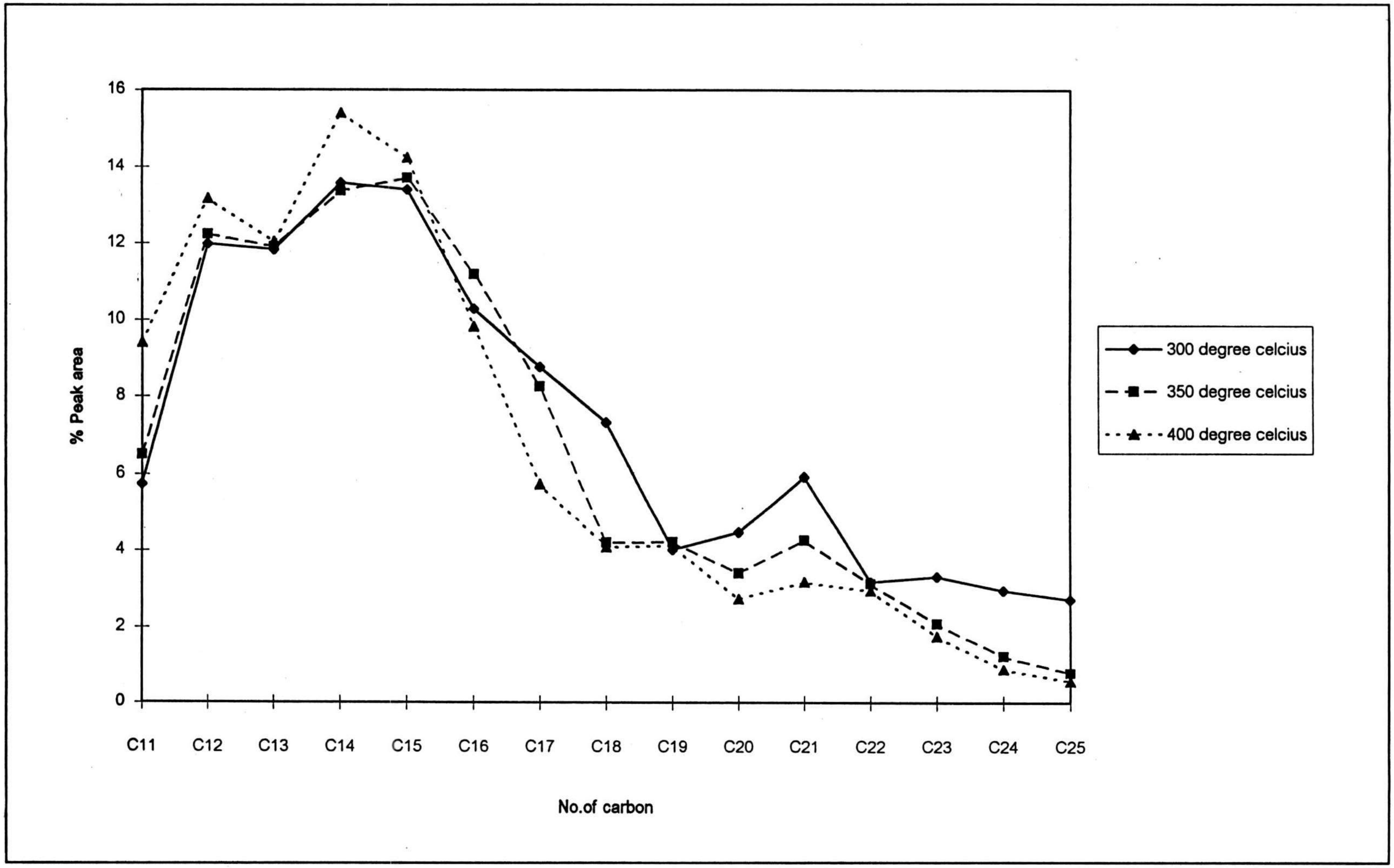

Figure 4.4 Composition trend of product from hydroisomerization as a function of temperature (second time)

4.2.3 Effect of hydrogen pressure

GC/MS, GC chromatogram and composition of products from hydroisomerization as a function of hydrogen pressure, using an otherwise constant set of reaction condition, are shown in Figures B3, B9 and Tables B5, B6, B13, B14, respectively. HDPE wax conversion and the composition trend as a function of above processing valuables are shown in Table 4.3 and Figures 4.5, 4.6, respectively.

As seen in Figure 4.5, 4.6,The product composition changes with changes in hydrogen pressure. The composition of products consist of C_{11}-C_{25} hydrocarbons with C_{11}-C_{16} being the main components. The main trend observed is that increase in hydrogen pressure has a beneficial effect on the yield of low molecular weight and higher molecular weight components. It can be seen that the general trends are analogous with those observed for the effect of reaction temperature i.e. with increasing hydrogen pressure there is a gradual increase in the yield of low molecular weight component, while decreasing in the yield of higher molecular weight component. However, as seen in Table 4.3 the hydrogen pressure range of 300-500 is indicated as satisfactory, but economic considerations dictate the use of minimum hydrogen pressure consistent with high conversion. In conclusion, a pressure in the vicinity of 300 psig could be considered optimal.

Table 4.3 HDPE wax conversion from hydroisomerization as a function of hydrogen pressure

Hydrogen Pressure (psig)	% conversion	
	first time	second time
100	-	-
200	-	-
300	100	100
400	100	100
500	100	100

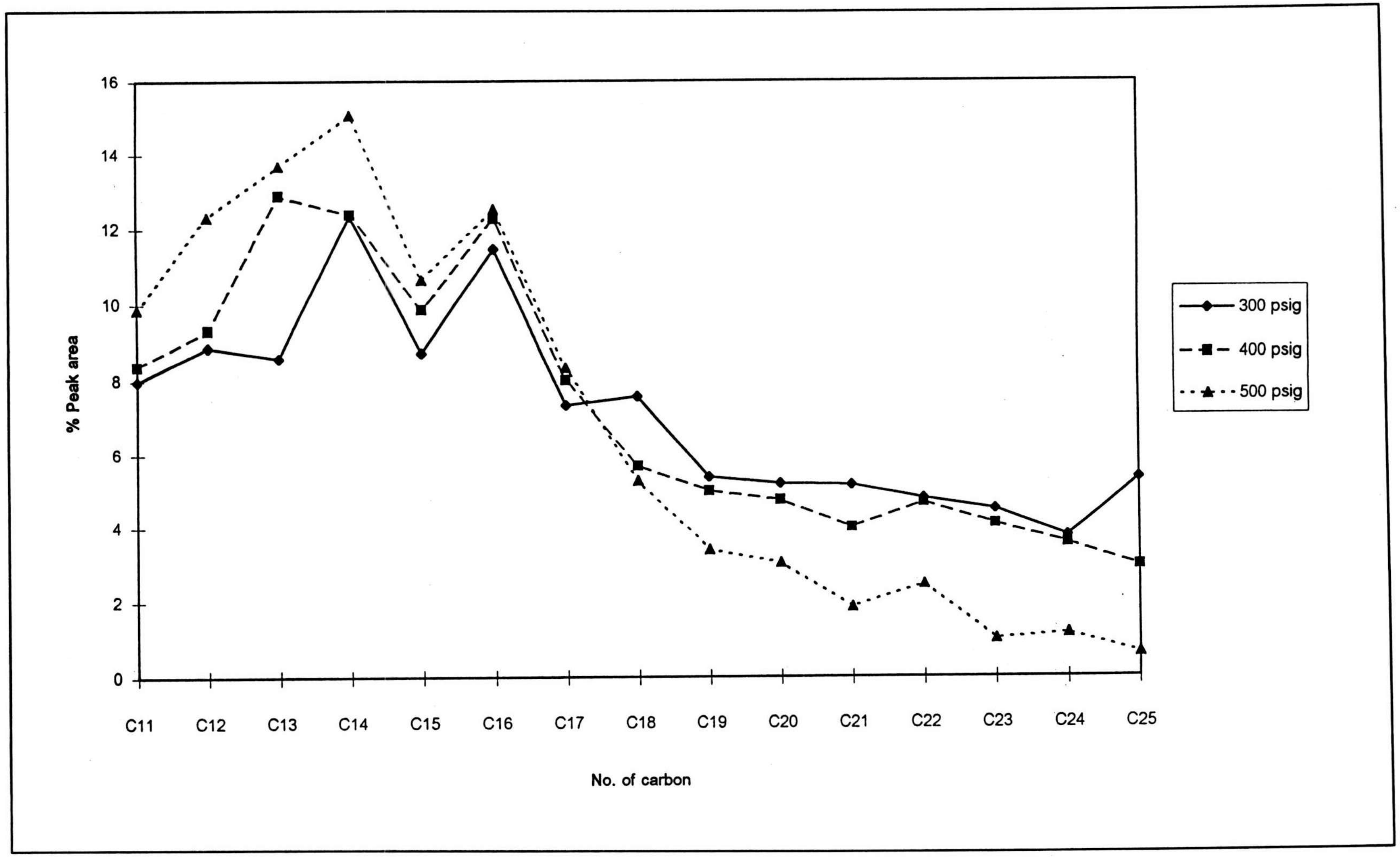

Figure 4.5 Composition trend of product from hydroisomerization as a function of hydrogen pressure

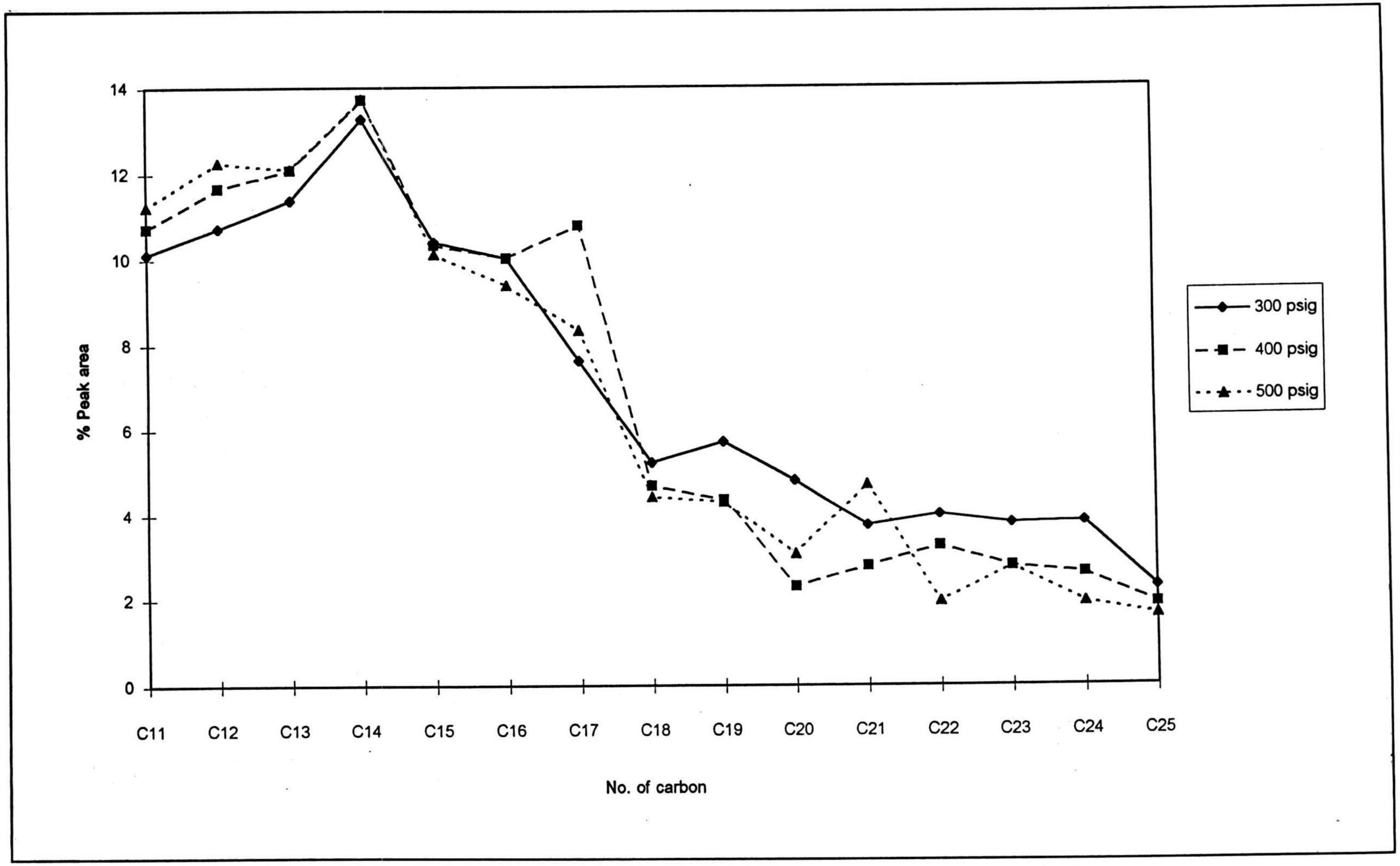

Figure 4.6 Composition trend of product from hydroisomerization as a function of hydrogen pressure (second time)

4.2.4 Effect of reaction time

GC/MS ,GC chromatogram and composition of products from hydroisomerization as a function of reaction time, using an otherwise identical set of reaction conditions, are shown in Figures B4, B10, B11 and Tables B7, B8, B15, B16, respectively. HDPE wax conversion and the composition trend as a function of above processing valuables are shown in Table 4.4 and Figures 4.7, 4.8, respectively.

As seen in Figures 4.7 and 4.8 the general trends are analogous with those observed for the effect of catalyst concentration, i.e. increasing reaction time only slightly changes the composition. The economic considerations dictate the use of optimum reaction time consistent with high conversion, therefore, the suitable reaction time, with constant other processing conditions, is 10 min.

Table 4.4 HDPE wax conversion from hydroisomerization as a function of reaction time

Reaction Time	% conversion	
(min)	first time	second time
10	100	100
60	100	100
120	100	100
180	100	100
240	100	100

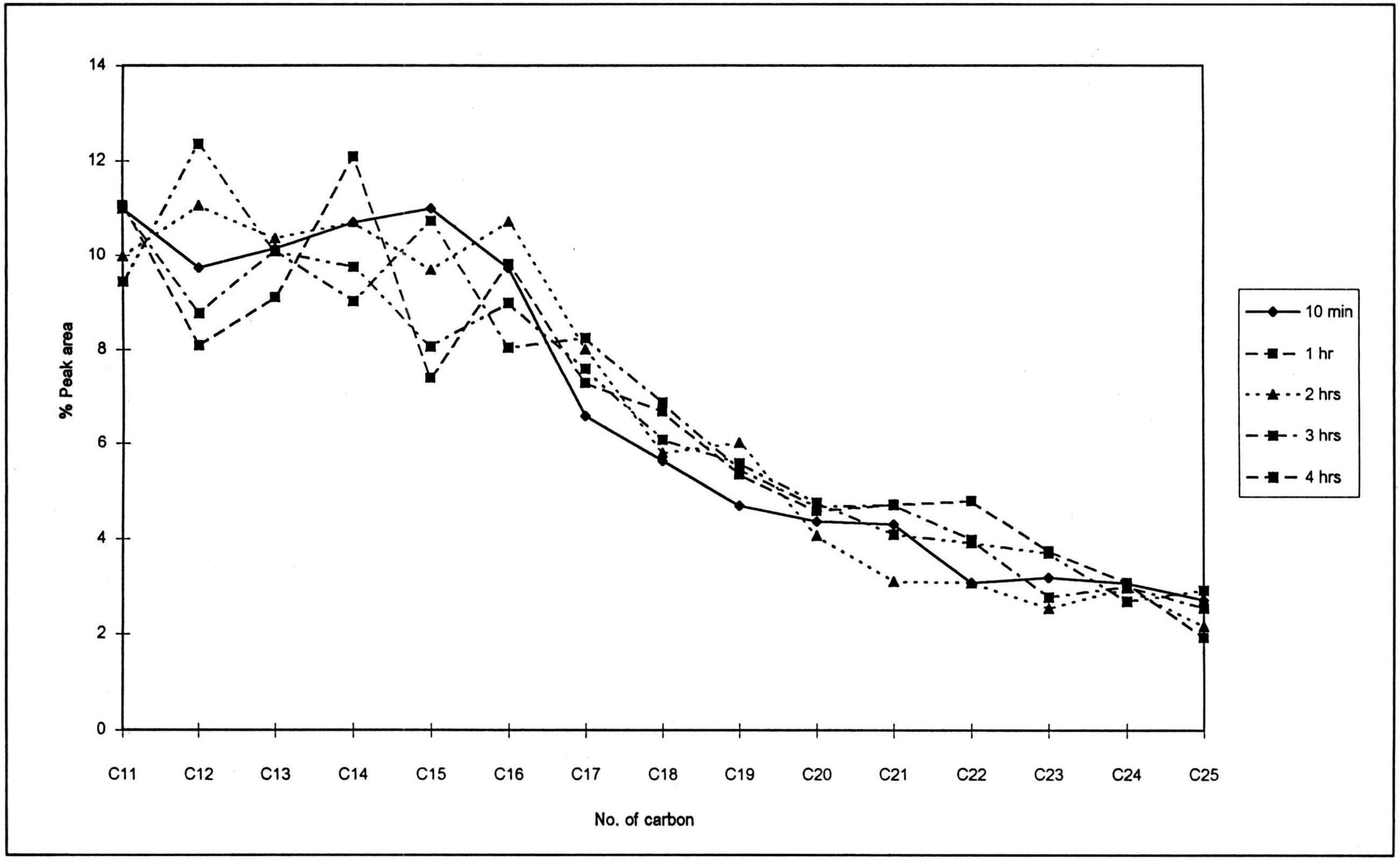

Figure 4.7 Composition trend of product from hydroisomerization as a function of reaction time

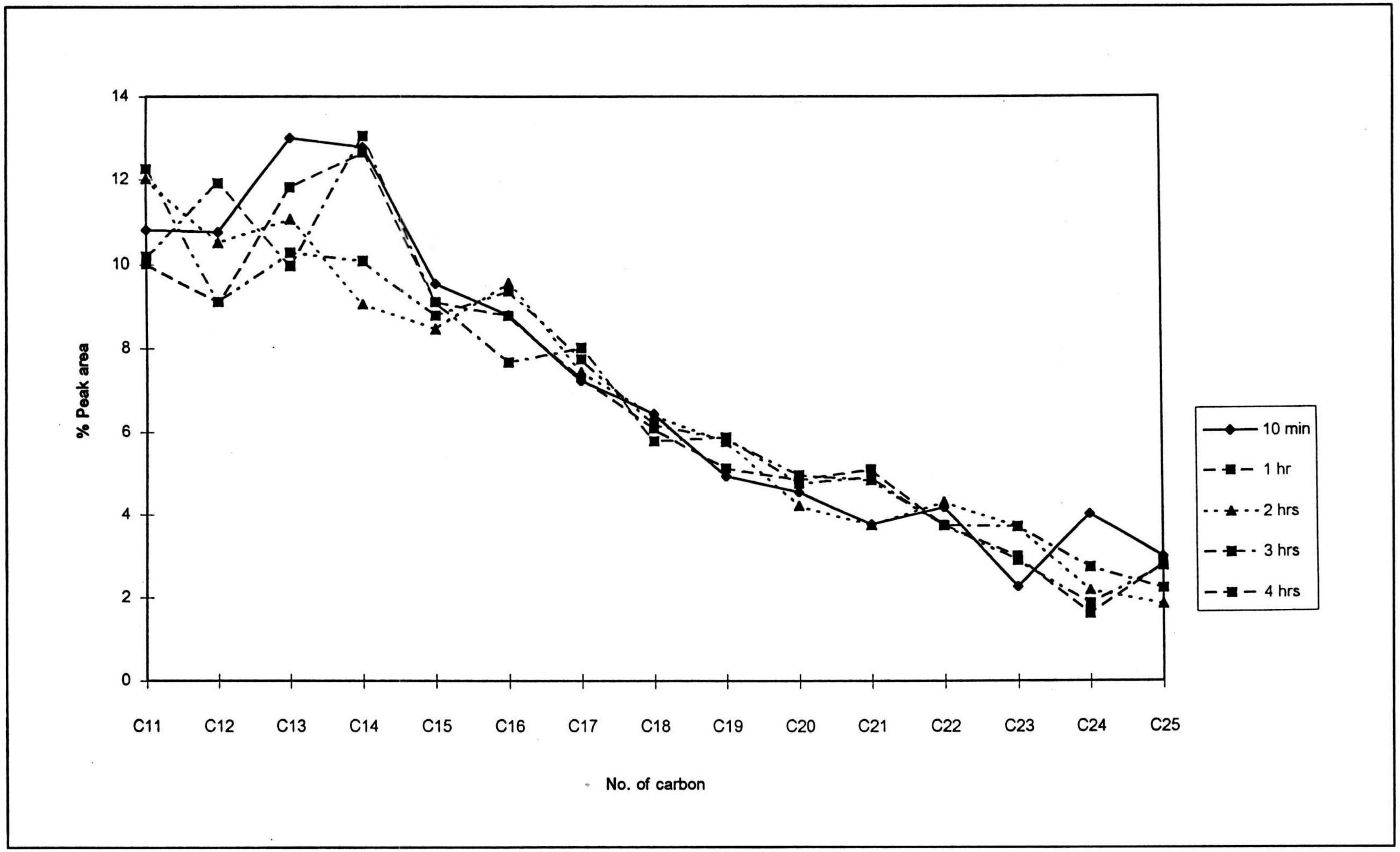

Figure 4.8 Composition trend of product from hydroisomerization as a function of reaction time (second time)

4.3 Determination of product properties

4.3.1 Spectroscopic properties

^{13}C NMR spectrum the product formed under optimum conditions product is shown in Figure A7. It shows C-H aliphatic at 10-40 ppm. This result demonstrated that the product consists of hydrocarbons.

4.3.2 Physical properties

The diesel properties of the product are shown in Table 4.5 In comparisons with the specifications of base diesel , this product is accepted to be low speed Diesel.

Table 4.5 The Diesel properties of optimum condition product

Properties	Method	High Speed Diesel	Low Speed Diesel	Product
Kinemetics Viscosity @ 40 °C, cSt	ASTM D 445	1.8 - 4.1	max 8.0	2.7716
Specific Gravity @ 15.6/15.6 °C	ASTM D 1286	0.81 - 0.87	max 0.92	0.8142
Color	ASTM D 1500	max 4.0	4.5 - 7.5	< 4
Pour Point, °C	ASTM D 97	max 10	max 16	13
Flash Point, °C	ASTM D 93	min 52	min 52	70
Sulfur Content, % wt	ASTM D 129	max 0.25	max 1.5	0.00
Distillation, °C	ASTM D 86	max 357	-	359
Calculated Cetane Index	ASTM D 976	min 47	min 45	63.8354

On comparing the product with the specification base diesel , it is seen that the advantage of the product from this work are the sulfur content and calculated Cetane Index. The product presents a high value of calculated Cetane Index and no sulfur content. The advantage of high calculated Cetane Index resides in decreasing of ignition delay and pollutants in exhaust emissions such as carbon monoxide, nitrogen oxide, hydrocarbon polyaromatics and particulates. The advantage of no sulfur content resides in decreasing particulate emissions and the effect of corrosive action.