



SYNTHESIS AND STUDY OF BIMETALLIC CATALYTIC SYSTEMS FORMED IN SITU BY ALUMINUM, 1, 2-DICHLOROETHANE AND Fe (III), Ni (II), Mn (II) CHLORIDES

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ABSTRACT

Novel bimetallic catalytic systems based on the catalytic complex (CTC) are synthesized from the reaction of Al metal and 1, 2-dichloroethane in the mode of in situ and modified with the metal chlorides of Mn (II), Fe (III), and Ni (II) in paraffin medium. The structures of synthesized catalytic samples were confirmed by bulk and surface characterization with FTIR, X-ray diffraction (XRD), thermal analyses (TG/DTG/DTA), dynamic light scattering spectroscopy (DLS) for determination the particle size and X-ray fluorescence microscopy (XRFM).

Keywords

CTC, CTC/FeCl₃, CTC/NiCl₂, CTC/MnCl₂, XRFM, TG/DTG/DTA, XRD.

Academic Discipline And Sub-Disciplines

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INTRODUCTION

As it is known [1, 2], the production of low molecular weight petrochemical complex olefins, particularly ethylene and propylene is carried out by the pyrolysis of raw hydrocarbons. Moreover, along with the desired products the liquid pyrolysis products which are formed in its composition with significant amounts are very valuable for organic synthesis of unsaturated and aromatic hydrocarbons depending on the feedstock nature and the process conditions. Consequently, the liquid products of pyrolysis (LPP) can be represented as an effective source of raw material for producing aromatic hydrocarbons (benzene, toluene, xylenes) and various petroleum resins resulting from the oligomerization process of unsaturated compounds in its composition. This makes the treatment of LPP to produce the above products are very up to date [3,4]. AlCl_3 is the most famous catalyst used for the synthesis of PPRs or its complexes with various compounds, including catalysts of the Ziegler-Natta type based on alkylaluminum. However, these catalysts do not have sufficient activity and stability and therefore are used in large quantities for carrying out the process [5-8]. To overcome all these drawbacks, catalytic complex (CTC) of the cationic type based on the reaction of aluminum metal and 1,2-dichloroethane was synthesized, and this type of catalytic complex showing a higher activity in comparison with other aluminum-containing catalysts in the processes of stable oligomerization and alkylation processes in the presence of LPP complex processing which is carried out with obtaining high purity of benzene fraction and PPRs.

Application of a new catalytic complex (CTC) for the LPP processing, along with obtaining high purity aromatics and PPRs as by product and used as coatings in the paint industry and road bitumen additives, provides a significant cost reduction of the overall process [9,10]. However, despite the fact that the synthesized catalytic complex based on aluminum dichloroethane as compared to other catalysts exhibit cationic activity greater stability in the test process involving its low molecular weight forms (PPR). To further enhancement of the catalytic behavior and high purity of PPRs there is a need for modification of these catalysts. For this purpose, the catalytic complex of Al-dichloroethane was modified with the transition metal chlorides. This is not only to improve the characteristic quality of the oligomerization products which is very important from the practical side, but at the same time to improve the efficiency of the pirocondensate recycling process [11].

This paper presents the results of the synthesis and the structure study of the catalytic systems CTC and its modified forms of Mn (II), Fe (III), and Ni (II) metal chlorides.

EXPERIMENTAL

2.1. Synthesis of the bimetallic catalysts

The synthesis routs of bimetallic catalysts were carried out in laboratory conditions in the mode of in situ in a three-necked flask equipped with mechanical stirrer, reflux condenser and thermometer. The influence of temperature, effect of reaction time, the effect of solvent nature, and the ratio of components including the molar ratio of Al:Me were studied for the process. The formed bi-metal complexes are designated as CTC/ NiCl_2 , CTC/ MnCl_2 , and CTC/ FeCl_3 . These bimetallic complexes were further calcined at 800°C in air to be analyzed by XRD method.

2.2. Characterization techniques

2.2.1. Dynamic light scattering Particle Size (DLS)

Particle size and size distribution of the catalytic systems were determined by dynamic light scattering (DLS) with particle size analyzer LB550, Horiba in 1, 2-dichloroethane as solvent at 25°C.

2.2.2. Thermal analysis (TG/DTG/DTA)

Thermal analysis (TG/DTG/DTA) was performed on thermograph Q-1500D MOM in the temperature range 20-800°C in flowing of air. The rate of heating oven was 10°C/min, with using calcined alumina as reference sample.

2.2.3. X-ray diffraction (XRD)

XRD patterns of the samples were recorded on an automatic diffractometer "D2 PHASER" Company Bruker (Germany) at room temperature. Diffraction patterns were obtained with Ni-filtered $\text{CuK}\alpha$ radiation ($\lambda = 0.15418 \text{ nm}$), monochromatic X-ray beam, and X-ray tube parameters with 35 kv and 25 μA .

2.2.4. FTIR spectra

IR spectra of the samples were recorded on a spectrophotometer "Bruker ALPHA FT-IR" in the range of 400-4000 cm^{-1} at 25°C. The spectra have been explained based on the literature [12-16].

2.2.5. X-ray fluorescence microscopy (XRF)

X-ray fluorescence microscopy XGT-7000 Horiba (XRF) was used to determine the elemental composition and the distribution of elements in layers of the catalytic systems. Accelerating voltage of 15 kV was used in determining the Al and Cl-elements, and for the elements Mn, Fe, Ni were determined by using X-ray tube voltage of 50 kV with a diameter of the incident X-ray beam 100 microns, the measurement time was 200 sec. for each sample. Before measurements, the samples were crushed to powder and pressed at pressure 20 tor/cm^2 .



RESULTS AND DISCUSSION

During the synthesis process of the catalyst, it was primarily studied the role of the solvent used in this process. Practically, it has been shown that by using benzene and its monosubstituted derivatives as solvent aromatic hydrocarbons are formed during the initial reaction period of aluminum chloride exerting a catalytic effect on the interaction of aromatic hydrocarbons with dichloroethane (DCE) contributes to their accumulation in the polycondensation system of polyphenylethyl and making it impossible to synthesis the catalytic systems. Using of normal hydrocarbon as solvent makes the synthesis conditions of the catalyst with a high yield is easy. By the chromatographic analysis of the gases separated in the synthesis process of the catalytic complex, it was suggested that the solvent molecules in the process act as a hydrogen donor, and further transformed into low molecular weight branched chain of hydrocarbons and after that are removed from the system. The compositions of the gases generated in the process are listed in Table 1.

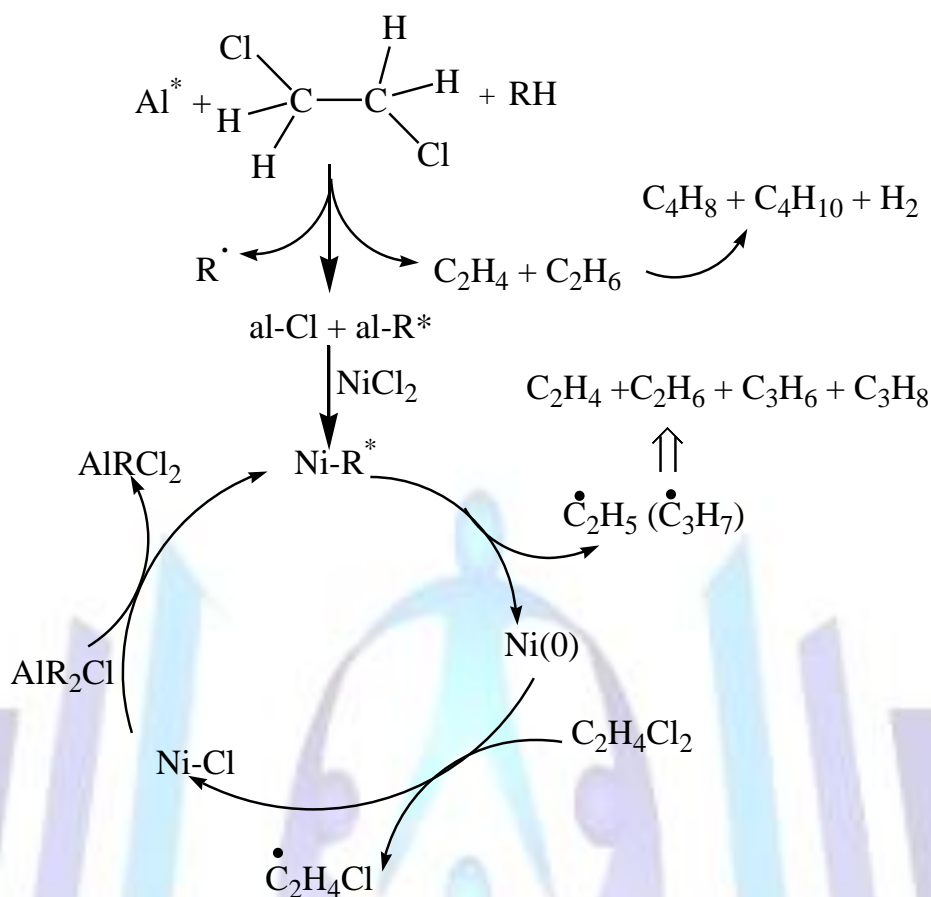
Table 1. The results of the chromatographic analysis of the gases generated in the synthesis process of bimetallic catalytic systems CTC and CTC/NiCl₂

Hydrocarbons	% wt.	
	CTC	CTC/NiCl ₂
Ethane, Ethylene	8.35	3.96
Propane, Propylene	15.33	10.13
Iso-butane	14.38	14.82
butylene	17.39	9.7
n-butane	7.5	4.22
Divinyl	4.82	2.28
iso-Pentane	8.3	17.42
n- Pentane	8.28	2.99
2.2-dimethylpropane	0.9	2.96
2.2- dimethylbutane	1.64	3.55
2.3- dimethylbutane	3.2	5.06
2- methylpentane	2.13	4.19
3- methylpentane	0.83	3.96
n-hexane	3.9	6.28
2.4- methylpentane	3.05	8.48

It is found that, the hydrocarbon composition of the gases generated in the synthesis process of the CTC and the bimetallic catalytic systems is identical, and the numbers of them are different. In bimetallic systems with transition metals unlike CTC observed a higher content of branched chain hydrocarbons and lower content of the hydrocarbon of ethane, ethylene and propane-propylene. This can explain the complex formation of bimetallic catalytic system by modifying the sample of CTC. The mechanism of formation of bimetallic complex can be represented as follows (Scheme 1):

The presence of carbon particles in the system can be explained by the reduction of methyl and ethyl radicals which formed during the reaction and by the presence of free carbon on the activated aluminum surface.

In the systems Al + DCE + heptane + Me_xCl_y full interaction of the components is directly related to the nature of the solvent and the molar ratio of the used components, temperature and reaction time.



Scheme1: mechanism of bimetallic complex formation

Al^* - Activated aluminum; al - $1/3\text{Al}$; R^\cdot - $\cdot\text{C}_2\text{H}_5$, $\cdot\text{C}_3\text{H}_7$ radicals, RH - solvent (heptane)

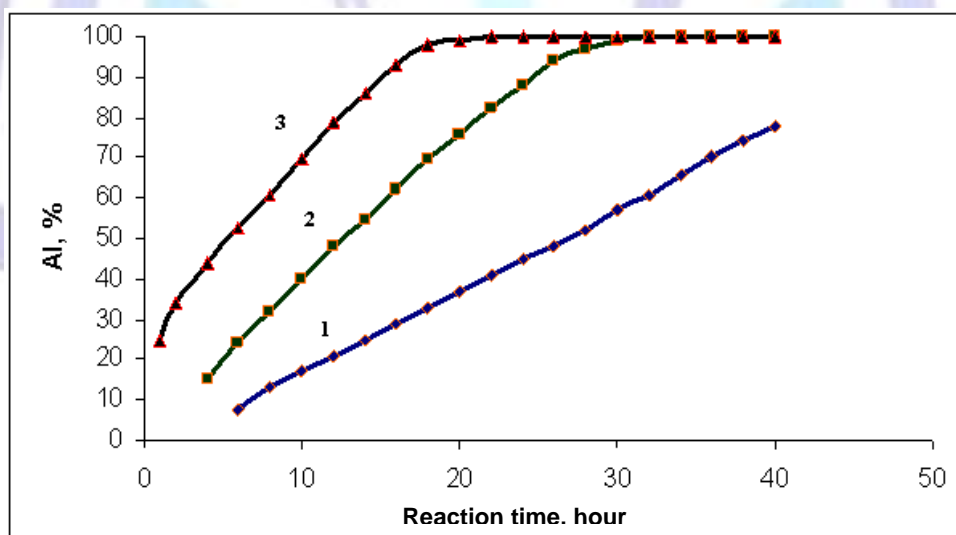


Fig 1: Graphical representation of the dependence of Al conversion on the reaction time at temperatures: 1-60°C, 2 - 70°C, 3-80°C.

As it can be seen from Figure 1, at low temperatures interval (60-70°C) for a total conversion of Al in the reaction requires more than 25-hours. When the temperature rises to 80 °C it is in turn accelerating the conversion of Al, respectively, the formation of the catalytic complex is completed at 20-22 hours. Given the boiling point of DCE, the reaction is carried out at temperatures not exceeding 80-85°C. The temperature below 60°C is not sufficient to activate the surface of the aluminum metal, and therefore its interactions with DCE.



Synthesis of bimetallic catalytic complexes was performed at molar ratios of Al to transition metal is equal 1:1 to 16:1.

At low ratios (Al:Me = 6:1, 4:1, 2:1, 1:1) although a complete consumption of aluminum metal process is occurred however, a part of the transition metal salt is precipitated or formed a mixture which remains in the system. At the ratios above 8:1, a complete and a simultaneous consumption of Al and the transition metal salts.

Dynamic light scattering Particle Size (DLS)

The method of DLS has been established that, the hydrodynamic sizes of all samples showed a non-symmetric distribution, and Table 2 shows the average particle size values of the bimetallic catalytic complexes. The CTC sample shows a comparison of the aggregation and impurities observed for the modified systems with metal chloride due to the addition of transition metals in the system which increases the size of particles and increases the degree of the aggregation.

Table 2. The particle size distributions and average particle size values of the bimetallic catalytic systems

Samples name	Diameter (nm) on 10%	Diameter (nm) on 50%	Diameter (nm) on 90%
CTC	11.9	27.4	44.2
CTC/NiCl ₂	35.7	87.7	144.4
CTC/MnCl ₂	37.4	88.2	142.0
CTC/FeCl ₃	41.7	95.7	156.4

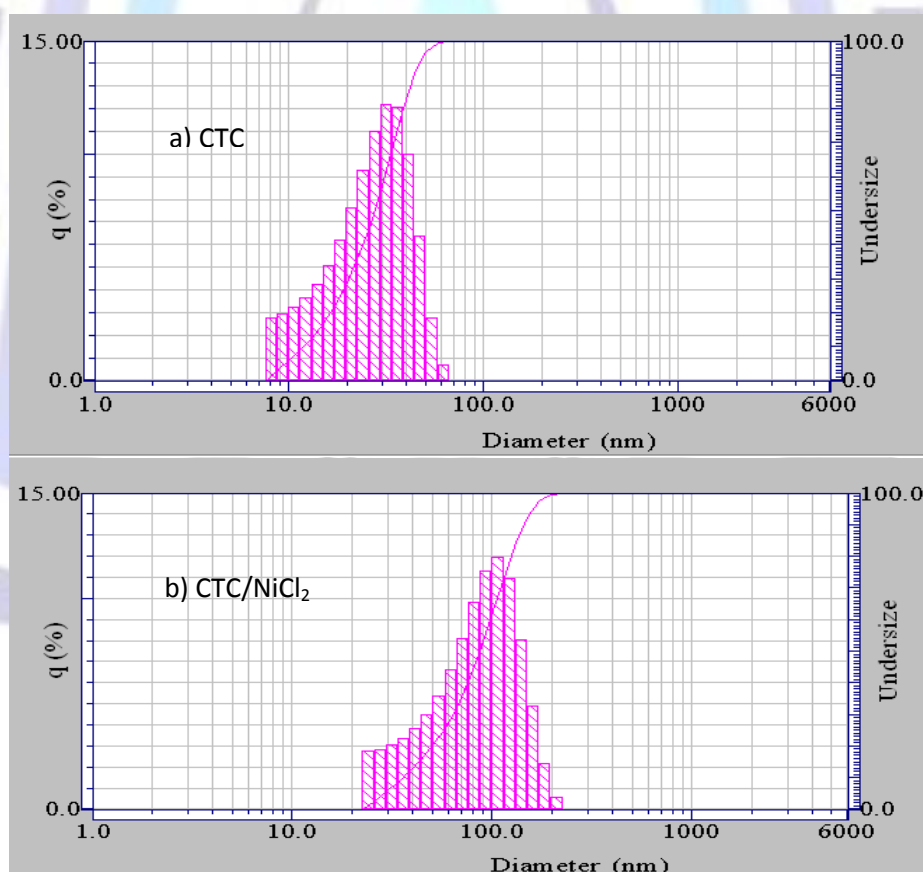


Fig 2: DLS spectra of the samples (a) CTC (b) CTC/NiCl₂

The results showed a wider size distribution and decrease in the particle size of the bimetallic catalytic complexes in the following arrange CTC/NiCl₂< CTC/MnCl₂< CTC/FeCl₃ depending on the composition of the synthesized bimetal catalytic system. Figure. 2 shows the different particle size distributions and different average particle size values of the samples depending on the composition as calculated by using the Stokes-Einstein equation:

$$D = K_B T / 6\pi\eta R \quad (1)$$



where k_B - is the Boltzmann constant ($1.38 \times 10^{-23} \text{ m}^2 \text{ kgs}^{-2} \text{ K}^{-1}$), T - is temperature (K), η - is viscosity (Pa s), R , the hydrodynamic radius.

Thermal analyses of the synthesized bimetallic complexes

The methods of TG/DTG/DTA studied the thermal properties of the synthesized bimetallic catalytic systems and the effect of Fe^{3+} , Mn^{2+} , and Ni^{2+} as transition metals on the formation of carbon-based materials in these catalytic systems. According to TG/DTG/DTA data it was observed that, the samples were evaluated in three stages in the temperature range of 20-800°C in flowing of air.

The first stage (up to 180-220°C) is observed for the thermal decomposition of the crystallized water molecules, The second stage (up to 340-370°C) is due to the thermal decomposition of dry solid residue CTC and its modified forms may be due to the collapse of aluminum compounds and these 3d metal complexes of organic and inorganic nature with simultaneous processes of sublimation of some compounds and the evaporation of solvent residue, accompanied by an endothermic effect. The third stage of weight loss at temperatures from 350-800°C is characterized by the oxidative destruction of relatively stable catalyst components - metal chlorides and carbon particles. This stage is accompanied by oxidation of carbon nanoparticles with an exothermic effect [17,18]. At 800 °C the modified chloride samples CTC and carbon particles are completely oxidized. From Figure.3 it can be seen that the decreasing of the maximum temperature (T_{Max}) in the bimetallic systems associated with the oxidation of carbon particles of the catalysts due to the increase of dispersion and structural defects of carbon particles resulting from the mutual influence of transition metal ions and carbon. The dispersion growth of carbon promotes the surface area of catalytic systems as well as its function to support the bimetallic centers.

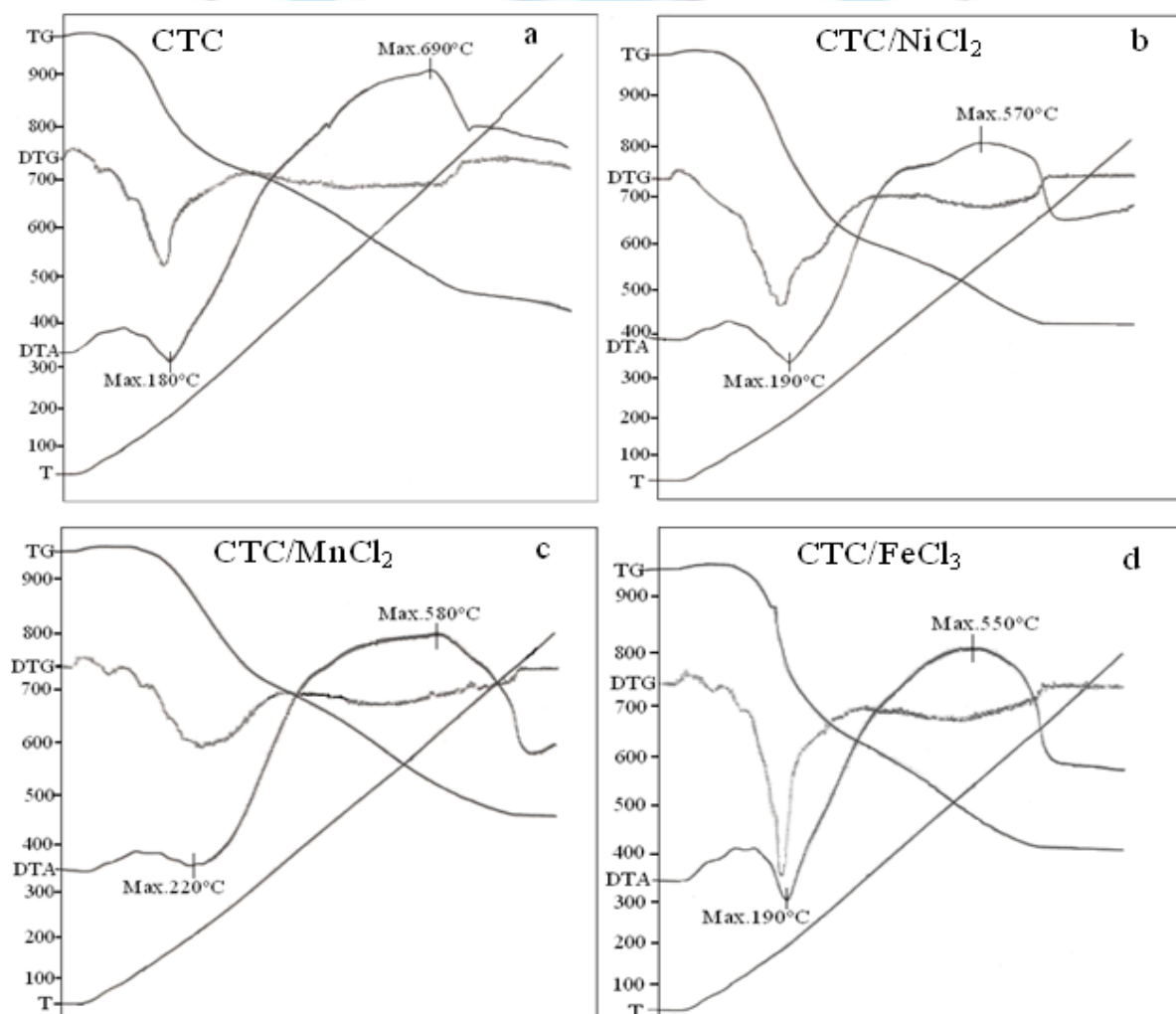


Fig 3: TG/DTG/DTA curves of the samples (a) CTC (b) CTC/ NiCl_2 (c) CTC/ MnCl_2 (d) CTC/ FeCl_3 .

XRD analyses

XRD patterns for the samples CTC, CTC/FeCl₃ dried at 100°C are shown in Figure. 4, The patterns showed that the samples have an amorphous structure at this thermal treatment but after calcinations at 800°C in air crystalline phases were obtained (Fig.5), the cubic phase of γ -Al₂O₃ was obtained for CTC and for CTC/FeCl₃ (PDF. 00-025-0063) at $2\theta=20^\circ, 34^\circ, 39^\circ, 40^\circ, 47^\circ$ and 64°), and also the phase of synthetic hematite Fe₂O₃ (PDF. 01-077-9927) at $2\theta=33^\circ, 36^\circ, 49^\circ, 54^\circ$ and 65° was formed for the sample of CTC/FeCl₃. The same behaviors were obtained for the samples of CTC/NiCl₂, CTC/MnCl₂ (not shown) treated at 100°C and those which calcined at 800°C.

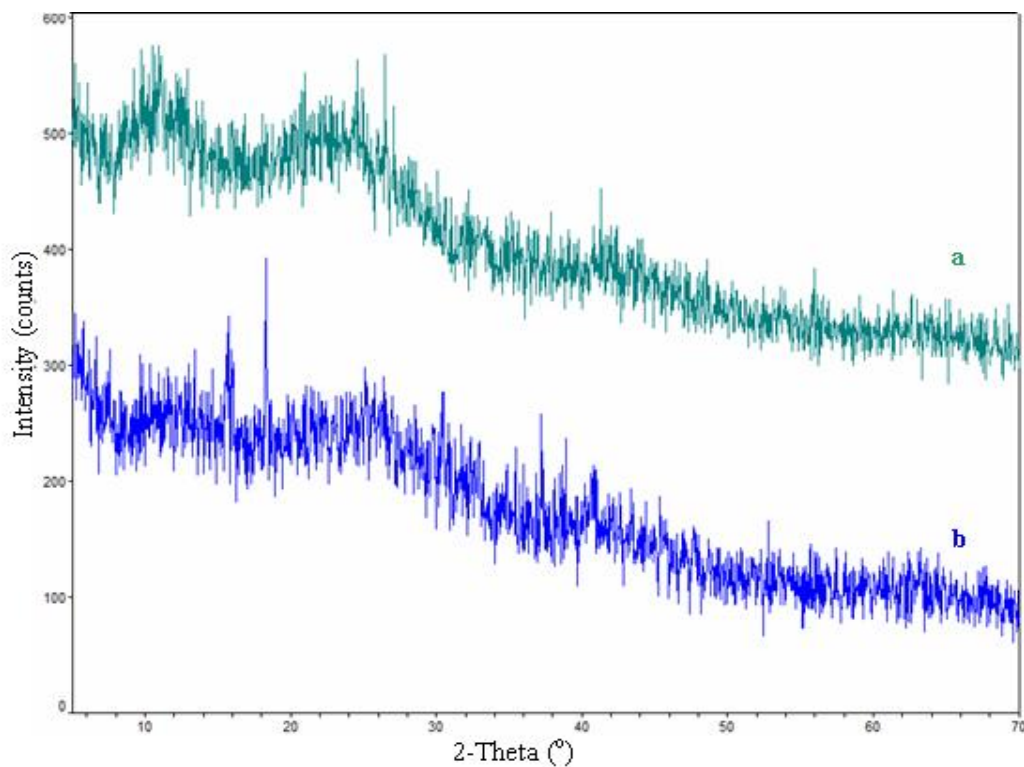


Fig 4: XRD patterns of precursor samples at 100°C (a) CTC (b) CTC/FeCl.

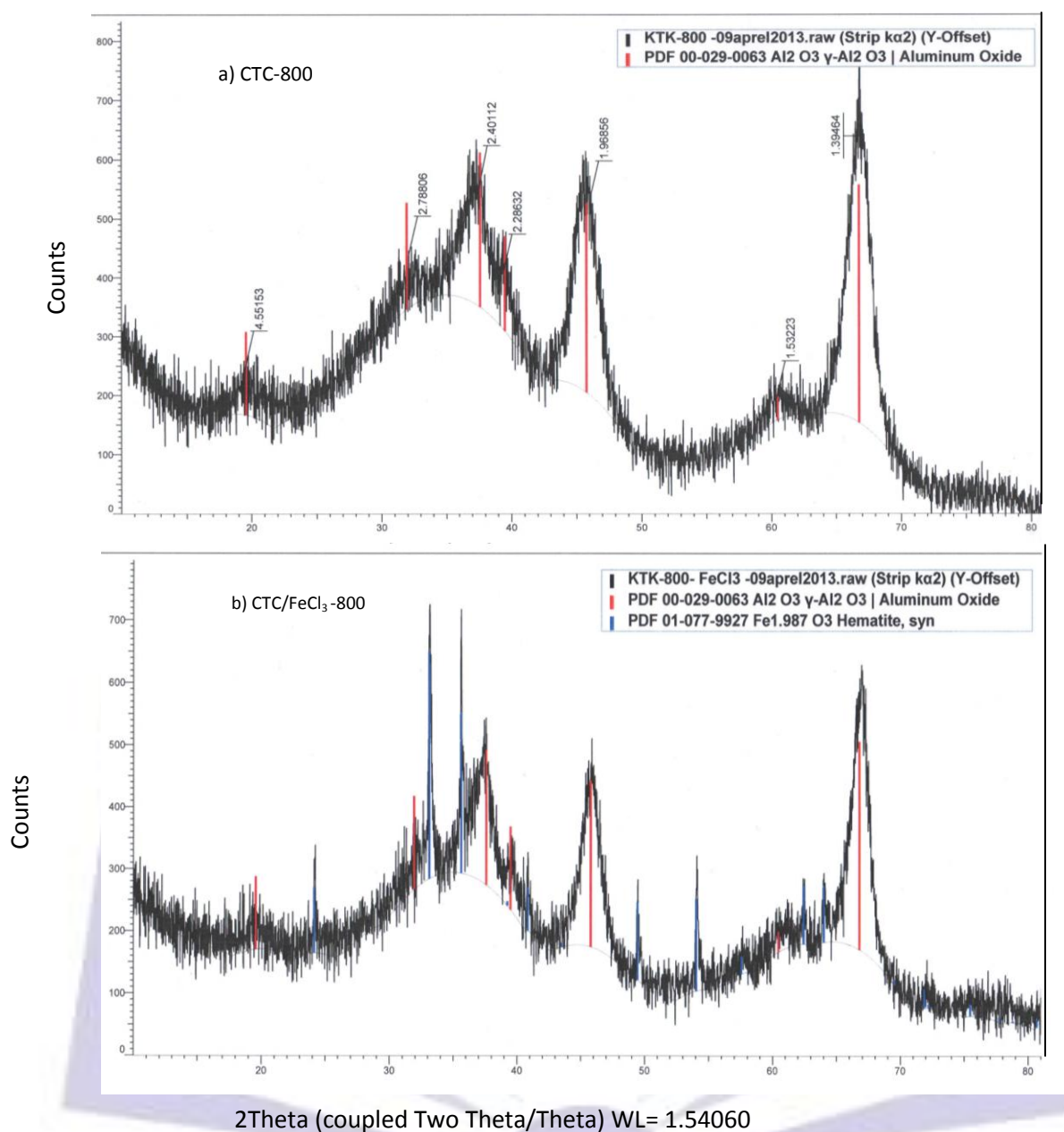


Fig 5: XRD patterns of the samples (a) CTC (b) CTC/FeCl₃ calcined at 800 °C

FTIR spectra

IR spectroscopy has been used to investigate the chemical composition of the catalytic systems consisting of clusters with a certain size in the liquid phase and to explain the method of complex formation.

IR spectra of the catalytic systems showed the 2800-3000 cm⁻¹ absorption bands belonging to the ν-CH stretching vibrations mode of the corresponding to CH₃ groups. In the areas of 1420-1490 cm⁻¹ and 1313.79-1369.74 cm⁻¹ absorption bands belonging to the bending vibrations mode of δ-CH bonds corresponding to CH₂ groups. As seen in the spectra of catalytic systems (Fig.6) the presence of stretching vibration mode of ν (OH) group in the 3300-3500 cm⁻¹ indicates the formation of bonds Me-OH, resulting from the crystallized water molecules and moisture formed into the catalytic systems in air, this was also confirmed by thermogravimetric analysis. The detected absorption bands for the sample of CTC in the range of 600-800 cm⁻¹ are related to the C- Cl - bonds. The IR spectra of the catalytic samples of CTC/NiCl₂ and CTC/FeCl₃ in this region showed three intense absorption bands. In the spectrum of CTC sample in the region marked by three bands of low intensity in the spectrum is of the same for the sample of CTC/MnCl₂ at 689.92 cm⁻¹ as an intense band. The absorption bands in the regions of 820-970 cm⁻¹ and 1230-1280 cm⁻¹, related to the C-H bonds are the same for different compounds [12, 13].



From previous work [15, 16] it is revealed that, the absorption bands at 490.06 cm^{-1} and 524.98 cm^{-1} are due to the stretching vibrations mode of the tetrahedral AlCl_4^- ions and at 584.72 cm^{-1} and $670\text{--}680\text{ cm}^{-1}$ band of Al_2Cl_7^- ions due to Al-Cl connection. The absorption bands marked at 405.36 cm^{-1} and 475.12 cm^{-1} correspond to Al-C bonds. Lack of the absorption bands in the region $600\text{--}620\text{ cm}^{-1}$ indicates that AlCl_3 in a molecular form is not present in the catalytic systems.

Based on the data of IR-spectroscopy, it can be concluded that the presence of C-Cl, Al-Cl, Al-C in the spectra, as well as methane and methylene groups indicates that during the reaction of Al with the DCE complexes ions are formed and belonging to the components reaction of Al_2Cl_6 , AlRCl_2 and AlR_2Cl (R - C_2H_5 , C_3H_7).

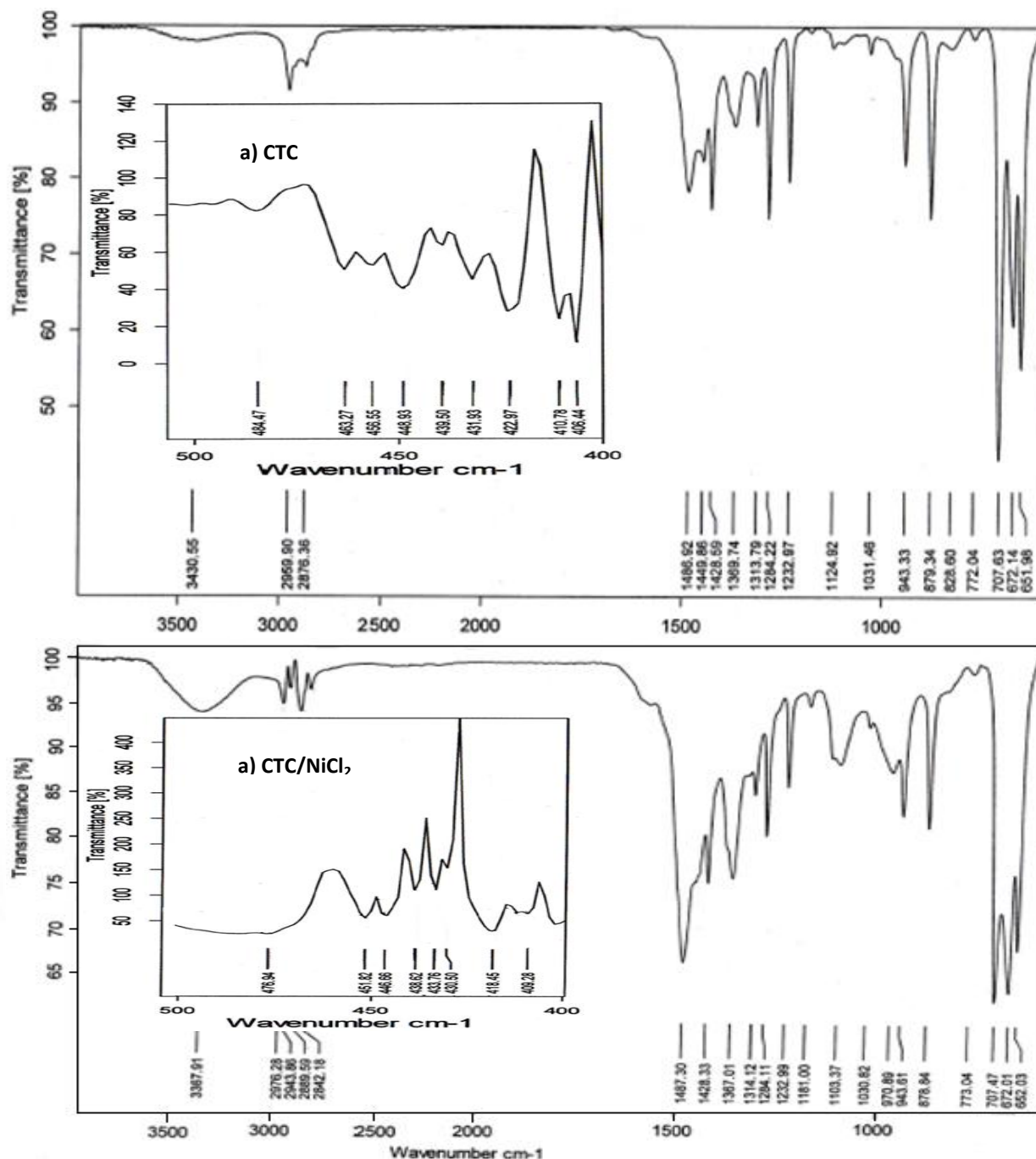
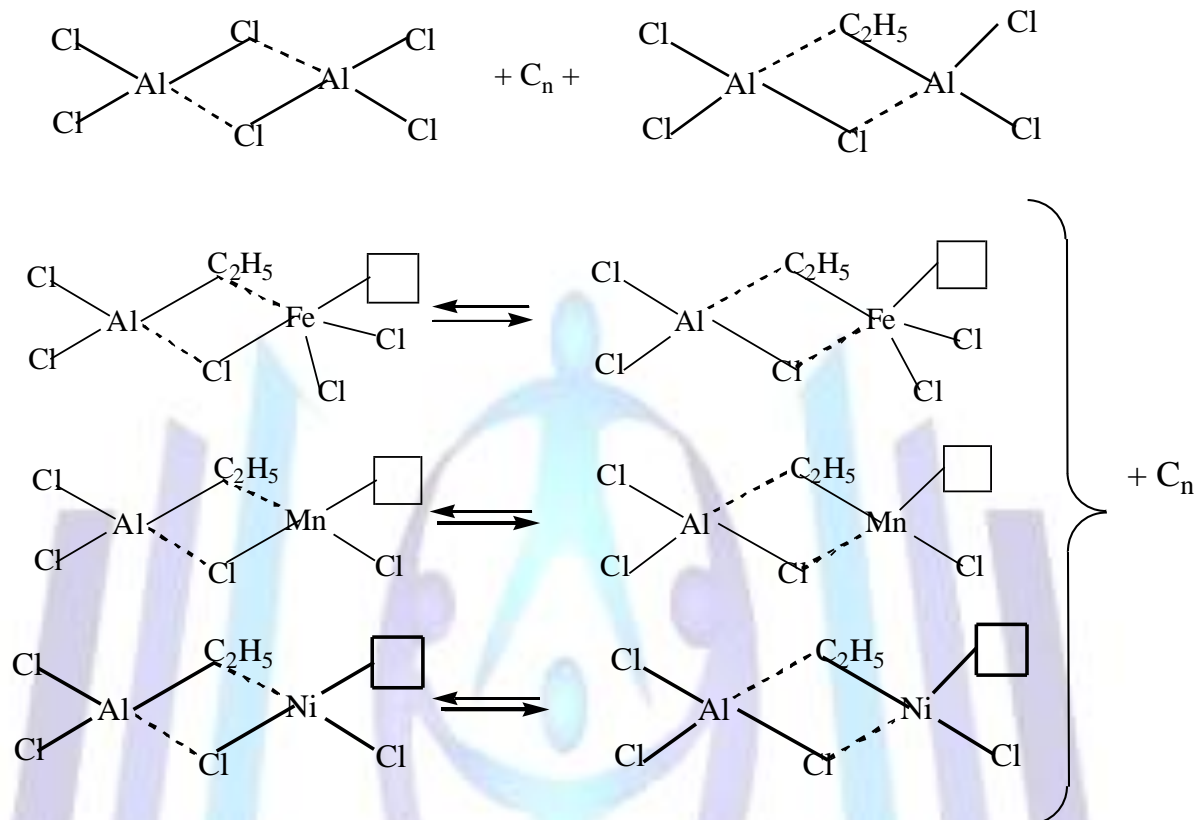


Fig 6: FTIR spectra of samples (a) CTC and (b) CTC/ NiCl_2

The presence of C-Cl bond proved the formation catalytic complexes from the reaction of AlCl_3 with dichloroethane (DEC), along with the above complex compounds forms $\text{AlCl}_4\text{C}_2\text{H}_4\text{Cl}^+$ ions. The assumed CTC and its bimetallic complexes can be represented as follows:



XRFM analysis

As discussed above, the catalyst CTC is the solid product of the reaction between aluminum metal and dichloroethane. The solid products which dried at 100°C represent a composite of inorganic salts and carbon particles. These solid residues of samples were characterized by XRF analysis as shown in Table 3. Data Listed in Table 3 showed that, the elemental distributions of these metals in the carbon matrix are significantly different for each sample (Fig.7).

Table 3. Percentage of elements in the samples and their distribution over the depth at 100°C

Samples name	Elements, wt%						Element distribution over depth, μm		
	Al	Cl	C	Mn	Fe	Ni	Mn	Fe	Ni
CTC	8.88	35.04	56.08	-	-	-	-	-	-
CTC/ MnCl_2	9.37	40.51	47.36	2.76	-	-	0.123	-	-
CTC/ FeCl_3	6.43	47.29	43.11	-	3.17	-	-	0.124	-
CTC/ NiCl_2	11.81	49.90	35.53	-	-	2.76	-	-	0.885

For the samples after thermal treatment at 800°C it was observed that the metal salts converted into oxides as example the elemental distribution of CTC sample is made up 99.02% of aluminum oxide and 0.98% of carbon. These elemental distributions of the bimetal catalytic systems after the thermal treatments of samples at 800°C provided the

formation of the metal oxides at higher temperature and this fact is agreed with the XRD diffraction analyses accompanied with the formation of γ - Al_2O_3 , hematite Fe_2O_3 , MnO_2 and nickel dioxide NiO phases.

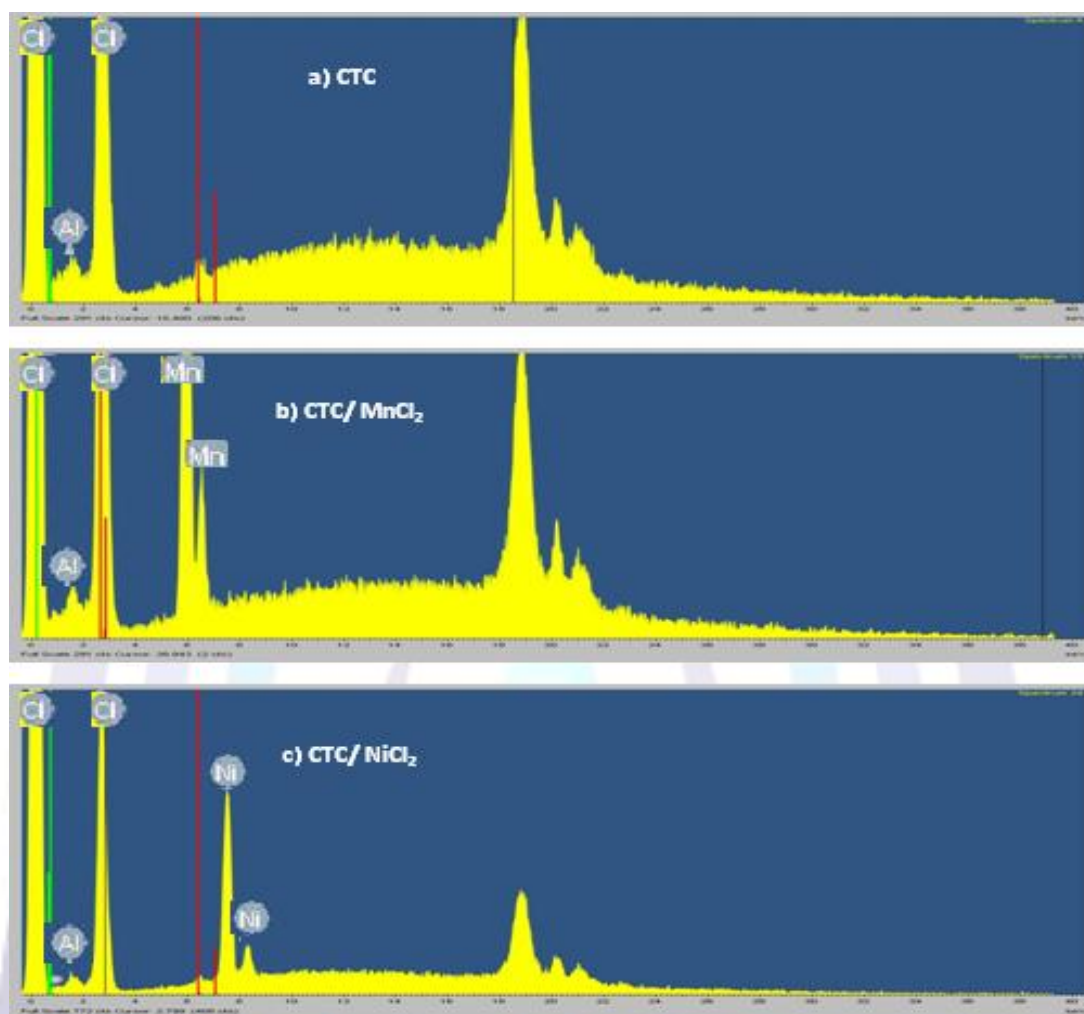


Fig 7: XRF analysis of the samples (a) CTC (b) CTC/ MnCl_2 (c) CTC/ NiCl_2

CONCLUSIONS

This work discussed the synthesis and study of the initial catalytic complex CTC and its modified bimetallic catalytic complexes of CTC/ FeCl_3 , CTC/ MnCl_2 and CTC/ NiCl_2 by the reaction of fine-dispersed Al metal with dichloroethane in paraffin medium in the presence of Fe (III), Mn(II) and Ni(II) chlorides as modifiers. This chemical-based synthesis route is briefly described, and the best conditions to synthesis these catalytic systems are concluded as follow: normal hydrocarbon is used as solvent as aromatic hydrocarbons with dichloroethane (DCE) accumulated in the system and makes the synthesis step is so difficult, the reaction is carried out at temperatures not exceeding 80-85 °C as below 60 °C it can not activate the surface of aluminum metal, the formation of the catalytic complex is completed at 20-22 hours and the best molar ratios of Al to transition metal is more than 8:1 to a complete consumption of Al metal and transition metal salts. the structures of the obtained catalytic systems have been confirmed by using different characterization techniques such as dynamic light scattering spectroscopy (DLS), thermal analyses (TG/DTG/DTA), X-ray diffraction (XRD), FTIR spectroscopy, and X-ray fluorescence microscopy (XRFM) which revealed the presence of aluminum, iron, Mn and Ni chlorides, and their nanoscale structures including the elemental distribution and mass thickness of the these elements over the layers. The XRD results also presented crystalline metal oxide phases of these catalysts in their inner structure after thermal treatment at 800°C. It can be assumed that the formation of these metal oxides is confirmed the presence of the metal chlorides as modifiers at the lower temperature 100 °C.

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Hikmet Jamal İbragimova. Doctor of Technical Sciences, Azerbaijan National Academy of Sciences, Institute of Petrochemical Processes. Vice director of Institute of Petrochemical Processes. 62 years old. He has more than 157 scientific and 19 patent. His Main area of activity: Development of new systems of catalytic cracking and pyrolysis, modernization of the existing ones, development of the technology for deepening of oil-refining and products of the catalytic cracking and pyrolysis satisfying by quality the world standards.



Kenul Murvat Gasimova. 29 years old. Working as researcher in Azerbaijan national academy of science, institute of petrochemical processes, PhD student in institute of petrochemical processes, Department of catalytic cracking and pyrolysis. She has more than 21 paper and theses in international journals and conferences. Her Main scientific achievements have been developed the theoretical foundations, synthesis and analysis of catalytic complexes and treating of pyrolysis liquid products.



Zenfira Maherram Ibragimova. 60 years old. Working as Fellow senior researcher in Azerbaijan national academy of science, institute of petrochemical processes, has more than 57 scientific works. She has innovated a technologic complex scheme with few stages and allows to produce ethyl benzene, naphthalene, oligomers of different purposes, solvents etc.



Irina Valentinovna Kolchikova. Nationality is Russian. 56 years old. Working as Senior Researcher in Azerbaijan national academy of science, institute of petrochemical processes, has 30 scientific works. She has been worked out the scheme of the pyrolysis liquid products refining using the catalytic complex of which has been commercialized.



Mai Mostafa Khalaf. Nationality is Egyptian. 29 years old. She received the master degree in physical chemistry from the University of Sohag, Sohag, Egypt, in 2010. working as assistant lecturer in chemistry department, faculty of science, Sohag University. PhD student in Azerbaijan national academy of science, institute of petrochemical processes, She is an author on more than 18 international scientific works. Her current research focused on scientific foundation for the effective heterogeneous catalysts preparation for the oil chemistry and for various nano-technological applications.



Aynura Enver Alieva. 30 years old. Working as assistant in Azerbaijan State Oil Academy, PhD student in Azerbaijan national academy of science, institute of petrochemical processes, has 4 papers.