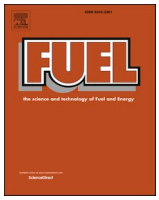
Fuel 334 (2023) 126735

Contents lists available at ScienceDirect

Fuel

journal homepage: www.elsevier.com/locate/fuel



Full Length Article

Catalytical thermal conversion of waste fishing nets for a higher added value energy products generation and caprolactam recovery

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ARTICLE INFO

Keywords:
Plastic wastes
Pyrolysis
Used fishing nets
Catalyst
ZSM-5
Y-Type

ABSTRACT

The ongoing eutrophication processes and water pollution caused by marine plastic waste are relevant ecological problem. Collected wastes are possible alternative feedstock for additional higher added value energy product generation. Moreover, caprolactam is the main compound of nylon-6 waste fishing nets and its recovery conserves natural resources, maximizes waste economic performance, and closes the circular economy loop of the fishing net industry. In order to contribute to the creation of circular economy, investigation of pyrolysis process and the effect of catalyst synergy with different temperatures on the formulated products has been performed. This work aims to analyse the used fishing nets (FN), using TGA-DTG-FTIR systems and mini pyrolysis plant. Experiments were conducted with Y-type zeolite and ZSM-5 as catalysts with a ratio of 1 by 3 and 1 by 8, respectively. Micro-thermal analysis using the TGA-DTG-FTIR system was processed for feedstock characterization purposes, which showed that the fishing gear has one minor decomposition peak around 200 °C, and the major one around 450 °C, with a total weight loss of 88 wt%. Pyro-oils analysis showed that the main fractions could be assigned to aromatic and aliphatic hydrocarbons, such as naphthalene, styrene, and toluene. Moreover, the recovery of caprolactam shows a possibility to obtain not only energy products, but also higher added value chemical derivatives. Temperature and catalyst synergetic approach influence for the recovered products investigation showed the optimum conditions as 700 °C for the highest purity and quality products generation. Based on the results, catalytic thermal treatment at 700 °C with Y-Type could be adapted as a promising technique for extracting of caprolactam with a high yield (96 %).

1. Introduction

In recent few decades, dramatically increased the demand for energy due to the continuous development of society, and the increase in population has led to the depletion of fossil fuel reservoirs [1]. Moreover, the rapid global society and population expansion increases total energy demand, creating a significant imbalance of energy consumption and production [2]. The combustion process of fossil fuels dramatically increases the amounts of carbon dioxide, nitrogen oxides, and sulphides, causing significant damage to the ecological environment and seriously threatening human health [3]. Reduction in greenhouse gas emissions by 50–80 % is the target set by the United Nations Climate Panel Committee by the year 2050 [4]. Thus, the consumption of renewable energy replacing fossil fuel has become an inevitable trend for the green development of human society [5]. Nowadays, many studies are focused on recycling for energy production or alternative pathways for promising energy carriers' recovery. However, the selection of energy-rich wastes is extremely wide, so an investigation of the feedstocks must be performed [6].

The plastics wastes on seashores are among the most dangerous oceans and water pool pollutants and one of the main factors causing the disruption of ocean ecosystems and marine bio-organisms [7]. Based on the recent studies, the total weight of the plastic in water pools is around 5.2 trillion particles, amounts to more than 260,000 tons [8], where these particles are produced from multiple sources related to wrong daily-life practices. The fishery activities are one of the main contributors to the production of the plastic waste and pollution, where approximately 700,000 tons of fishing gear are discarded or lost in the water pools every year, and annually this gear captures and kills on average 130,000 whales, seals, and sea lions [9]. Nevertheless, this type of waste interrupts the movement of ocean animals, leading turtles and dolphins to get entangled in the nets and die [10].

Energy recovery from plastic waste is another solution that is classified as a simple method and does not require much effort and

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https://doi.org/10.1016/j.fuel.2022.126735
Received 12 July 2022; Received in revised form 31 October 2022; Accepted 10 November 2022
Available online 17 November 2022

treatments besides simple costs compared to the cost of return [11]. The pyrolysis process is one of the promising energy conversion technologies used for that purpose and is characterised by its simple and environment-friendly method. This process produces three types of products namely pyro-oil, gas, and char, depending on the operating conditions [12]. Based on that, several investigations were performed to study the pyrolysis behaviour of FN using a micro-scale thermogravimetry analysis for polymers recovery [13,14]. As could be seen, FN can decompose into additional energy products and beta-caprolactam monomers, which is the main compound used for the preparation of nylon-6 polymeric fibres [15]. The recovery of these monomeric chains could lead to conserving resources, reducing waste, and reducing environmental burdens, thus, finding other sources for its production with less expensive [16].

Moreover, the effect of ageing in seawater during the pyrolysis process was also investigated by E. Jang [17]. It was assumed that ageing process changes the chemical structure, kinetic parameters, and decomposition temperature. The partial decomposition of peptide bond was the main change in the investigated feedstock during the ageing process. Moreover, R. Skvorčinskienė et al. [14] investigated the main thermal characteristics of fishing gear. Scientists determined that the most significant decomposition peak during the micro-thermal analysis is in the range of 400–440 °C, depending on the heating rate.

The most considerable influence for the polyamide's pyrolysis products generation could be defined by the temperature. A. M. Pannase et al. [18] have investigated the temperature and heating rate impact on pyrolysis oils composition and the yield. Scientists determined that with a lower heating rate, the obtained pyro-oil has a lower density and a higher volume compared to a high heating rate. The higher maximum temperatures and heating rates lead to a higher yield of pyro-oil. The obtained liquid pyrolysis products range from 85 to 88 wt%, while the gaseous products detected about 12–14 wt%, and only a negligible amount of char was obtained. During the studies was investigated that the quality and quantities of the formed products are decent and the technology shows a promising pathway. However, to increase the amounts and the quality of the products catalytical pyrolysis process could be applied.

Author R. K. Mishra et al. [19] investigated the different catalyst concentrations and the influence on the oil quality at the maximum temperature around 900 °C. It was estimated that the feedstock to the catalyst of 8:1 improved the characteristics of the oil by reducing the viscosity, oxygen content, density, and increasing gross heating value.

The fishing nets catalytic thermal conversion into valuable, high-quality energy products was widely described by Chaidan et al. [20]. The authors investigated used fishing nets thermal treatment over calcined scallop shells for the recovery of ε-caprolactam monomer. It was determined that the catalyst increased the yield of purposive products (ε-caprolactam) up to 66 wt%. at 410 °C with 2 min of resident time. Another catalyst applies for the fishing nets thermal decomposition and higher quality products formation is difficult to find. However, the review shows that fishing nets pyrolysis is relevant, the feedstock is applicable in the pyrolysis technologies and the further analysis must be performed.

Based on the literature review it is clear that the thermal treatment is promising alternative way for the additional energy carriers' and higher added value products generation from the wastes. These refuses could be collected from the seashores reaching the pollute-less and cleaner environment. In this study, the fishing nets were used as a feedstock for pyrolysis tests and chemical composition of formed volatile products analysis. The weight loss mechanism and temperature range were analysed by thermogravimetry, while the composition of formed products was evaluated by coupled thermogravimetry-FTIR system. Moreover, the investigation of pyrolysis process over different ZSM-5 and Y-Type was performed. To analyse the physical products using GC/Ms and GC/TCD pyrolysis test in laboratory scale mini plant was performed under the different temperatures (900–700-500 °C) and the same ZSM-5 and Y-Type catalyst.

2. Experimental methodology

2.1. Material

Used fishing nets were collected for all experiments in Melnragė beach, Klaipėda city, Lithuania. Firstly, the feedstock for thermal treatment was washed to remove the highest part of sand and other abrasive particles. After that, the fishing nets was dried in a dryer according to ISO 579, and copped to obtain a homogeneous fraction.

The catalyst was prepared using chemical and calcination processes to be ready for using directly. Also, ZSM-5 catalyst has the following specifications: SiO_2/Al_2O_3 molar ratio (38), shape (column pelletized), pore volume (≥0.25 ml/g), dimension (Φ2 × 2–10 mm), bulk density (0.72 kg/l), specific surface area (≥250 m^2/g), crushing strength (≥98 N/cm^2), attrition (<1 wt%), pore size (~5 Å), binder type (Pseudo-Boehmite-30 wt%), while the Y-Type-RE2 catalyst could be described as follow: SiO_2/Al_2O_3 molar ratio (>5), Unit Cell size (<2.453 nm), specific surface area (≥578 m^2/g), Particle Size Distribution D50 (<6 μm), bulk density (0.68 kg/l).

2.2. Analysis of the selected feedstock

The primary characterization and feedstock investigation were maintained using ultimate and proximate analysis. Those analyses were performed in accordance with LST EN 14918 for the LHV by an IKA C5000 calorimeter, LST EN 15148 for the volatile content, LST EN 14775 for the ash content, LST EN 14774-1 for the moisture content, and LST EN 15104 for the CHNS content by a Flash 2000 CHNS analyser. In addition, the heavy metals and minerals content were investigated following LST EN 15411: 2011 and LST EN 15297: 2011 using Induced Plasma Optical Emission Spectrometer (ICP-OES) Optima 8000. The results are presented in Table 1.

The functional groups' analysis in the waste FN sample was performed using FTIR spectroscopy, and the spectra results are depicted in Fig. 1. As could be seen from the results, the FTIR spectra contain peaks, particularly in the range of 3600–3100 cm^{-1} and 1600–1500 cm^{-1} representing secondary amine (C—N) bond in nylon-6, aliphatic C—H group (PP, PE, nylon-6) at 3000 cm^{-1}, carbonylic C═O group at 1700 cm^{-1}, CH_3 symmetrical bending at 1500 cm^{-1}, and C—H group in the range of 1200–1100 cm^{-1}.

2.3. Chemical and micro-thermal analysis by TG-FTIR system

Thermal degradation of the selected feedstock was investigated using a thermogravimetric analyser (Netzsch Jupiter F3, Germany) combined with Fourier transform infrared spectrometer (Bruker Tensor 27, Germany) system (TG-FTIR). All experiments were maintained with 15–20 mg of each batch in anoxic ambient using nitrogen with a 60 ml/min flow rate. Based on the literature review, the catalytical pyrolysis

Table 1
Main characteristics of feedstock used in the test.

Parameter	Used Fishing nets
Carbon, wt.%	53.20 ± 0.15
Hydrogen, wt.%	8.17 ± 0.04
Nitrogen, wt.%	7.44 ± 0.02
Sulphur, wt.%	<0.01 ± 0.00
Chloride, wt.%	1.14 ± 0.03
Oxygen, wt.% (diff.)	25.53 ± 0.08
Moisture, wt.%	1.8 ± 0.04
Volatiles, wt.%	96.54 ± 0.31
Fixed carbon, wt.%	0.92 ± 0.01
Ashes, wt.%	0.74 ± 0.01
LHV, MJ/kg	25.68 ± 0.1

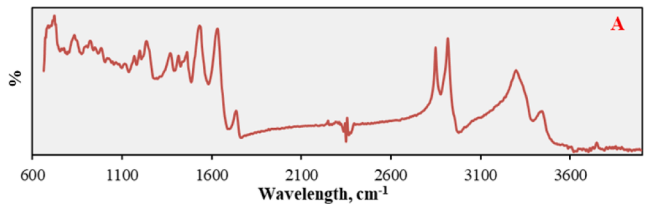

Fig. 1. FTIR analysis of the FN sample.

experiments with Y-type Zeolite and ZSM-5 catalyst in the ratio of 1:3 and 1:8 by catalyst: feedstock, respectively, were performed using the same conditions [16]. The experiments were maintained in the temperature ranges of 40–850 °C. DTG curves were recorded by derivation of TGA curves followed by an indication of each sample's maximum thermal decomposition zone.

2.4. Pyrolysis experiments at laboratory scale bench

The pyrolysis experiments using the fishing nets samples were performed in a laboratory-scale mini pyrolysis plant. Based on the discrepancies between the results provided by the different authors in the introduction section, it was determined to analyse the distribution of the products up to the maximum temperatures of 500–700-900 °C with 20 min of isotherm at the maximum temperature. The heating rate was set to be around 20 °C/min, based on our previous publication [16]. A detailed laboratory-scale bench scheme is depicted in Fig. 2. The weight of the sample used for the pyrolysis test was 100 g per load. Nitrogen is used as an atmosphere creating gas with a flow of 3 l/min. The pyrolysis plant consists of the main reactor chamber connected with a refinery at its end, with small holes, up to 2–3 mm, allowing the formulated gaseous and liquid products to exit, keeping the char fraction inside the reactor. The gathered liquid products are set down in the storage at the end of the reactor, while the gaseous products pass through a piping system and start colling, purification, analysis, and collection. The purification process of the formulated gaseous products was maintained simultaneously by passing through five washing bottles loaded with isopropanol. The composition of the purified gasses was analysed in continuous mode using VISIT 03H gas analyser.

In the case of other light C_nH_m present, the CH_4 concentration values might have higher inaccuracy due to the IR method. Therefore, to be more specific, from time to time, the GC measurements were applied. Collected liquid and gaseous products were analysed using GC/MS (Agilent 7890A) system, and the main compounds were determined using the NIST database. The used carrier gas flow in GC/MS system is 1.5 ml/min, while the main investigation interval is 30–600 *m/z*. The column of the used system is HP-5MS with a (5 %-phenyl)-methylpolysiloxane filling.

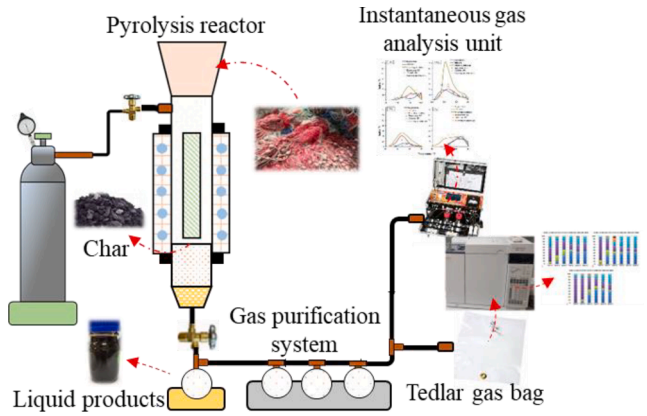

Fig. 2. Schematic view of mini pyrolysis plant.

Every experiment was repeated at least three times, and the deviation does not exceed 5 %, showing the reliability of the results.

2.5. Decomposition mechanism of waste fishing nets

The investigated pyrolysis decomposition mechanism was explained based on the liquid products analysis results, and the process parameters. The visualization of the process is depicted in Fig. 3. It was observed, that the material decomposed on three main stages. The first stage could be assigned to moisture evaporation and additives (PP, PE) decomposition from the feedstock. The sample was dried based on the methodology mentioned before, but the chemically bounded moisture stays in the feedstock, leading to the evaporation only at the higher temperature, when the hydrogen bond breaks. In the second stage broke of the chemical and mechanical bonds between the different polymers and atoms could be occurred, which is caused by the heat flux. Basically, three types of decomposition reactions could be noticed: side group reactions, polymeric chain scission, and recombination. Temperature increasement in this stage leads to the polymeric chain, including random and end chain scission, decomposition and forms free radicals and small molecules. For these reactions, the major role takes for the end chain β-scission, which need less energy than a random chain degradation, while the increased temperature leads to the start of chaotic reactions and random chain β-scission reactions present. It has to be mentioned, that end and random chain β-scission reactions were responsible for the fractions around C6–C20 of the liquid products, while the radical recombination reactions and intramolecular hydrogen shift were responsible for formation of olefin [21]. When the temperature is high-enough to decompose the bonds into the feedstock, and the major part of volatile fractions evaporates, the char formation (stage III) process begins. Generally, slow heating rates and long residence time into the reactor increases the char formation process [22,23]. Formed char is mixed together with inorganic molecules and contaminants from the feedstock, but based on the literature, most of the time, the amount of this fraction is negligible (<1 wt%) [24].

3. Results and discussion

3.1. TGA-DTG data analysis

Fig. 4 shows the results of TGA-DTG obtained during pyrolysis of the selected feedstock. The fishing nets have a minor decomposition zone around 200 °C due to the evaporation of contaminants and chemical residue from seawater. Also, it could have led to the decomposition of other polymeric chains with the primary polymer (nylon-6), such as PP and PE [15]. Moreover, the significant degradation has occurred around 440 °C. This decomposition could be explained by the linear condensation and heat penetration of the polymeric chains in nylon-6 (–NH-CO- bonds, recurring amide). As a result, the major part of long polymeric chains was decomposed and converted into pyrolysis products, including volatile liquids (paraffin, aromatic hydrocarbons, etc.), gases (H_2, CH_4, monomers), and solid char particles [14]. As reviewed in the literature, the highest degradation point of these polymers varies from 400 °C to 460 °C depending on the heating rate [25].

Catalytical micro-thermal analysis showed that the catalyst significantly increased the thermal resistance of samples decomposition in terms of total weight loss. This effect could be explained by the inability to decompose ZSM-5 and Y-type catalysts, which remain as a residue during the pyrolysis process. Thus, the residual mass in TGA experimental data must be recalculated to evaluate accurate results [26]. Fig. 4 A shows the recalculated results based on the amount of catalyst. The analysis of the assessed results indicates that adding of catalyst did not affect the weight loss and thus the performance of thermal decomposition itself in the main decomposition areas. The DTG curves (Fig. 4 B) show a sharp peak in the fishing nets decomposition area (about 440 °C), so it does not significantly change the features but the formulated

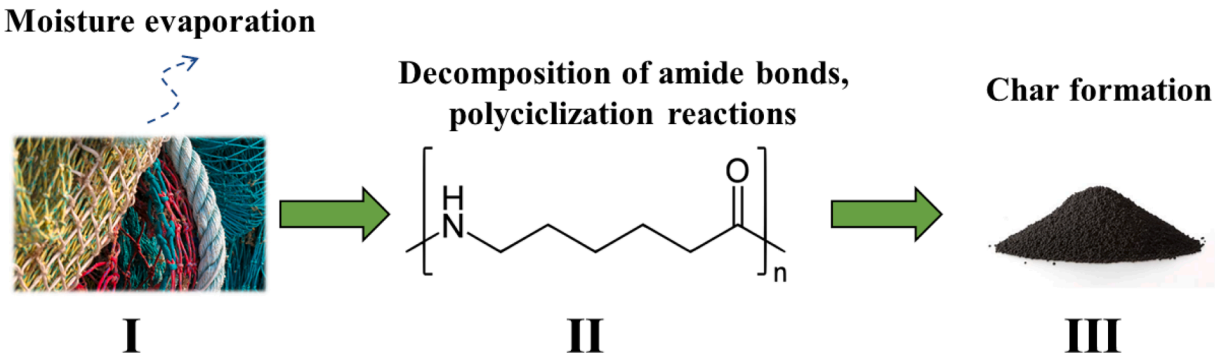

Fig. 3. Mechanism of waste fishing nets pyrolysis.

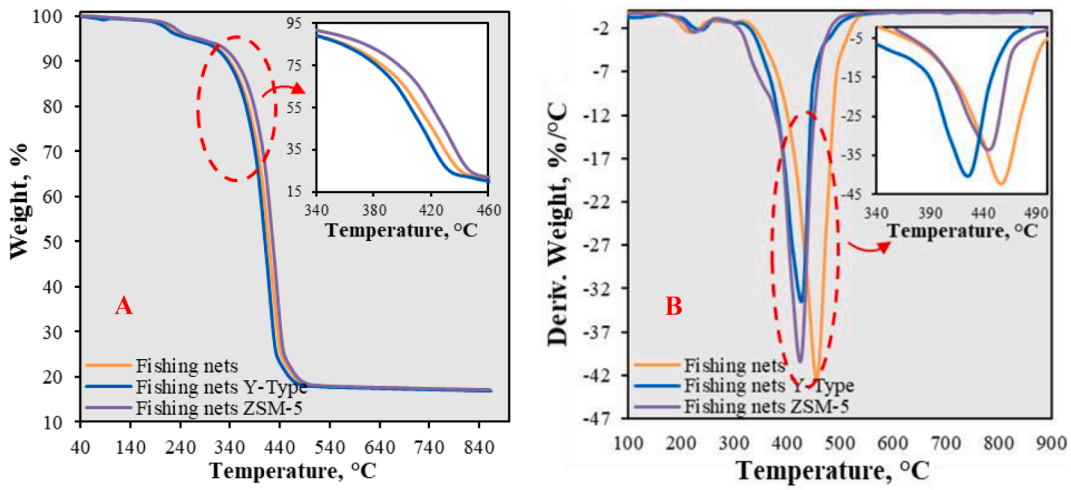

Fig. 4. TGA-DTG analysis of the used fishing nets (A-B) with and without catalyst.

chemicals products.

3.2. Gaseous products analysis by TGA-FTIR

In order to evaluate the volatile products during the micro-thermal analysis, the combined TGA-FTIR systems have been used. Fig. 5 shows 2-3D FTIR evolved gas spectra at the maximum decomposition peak during pyrolysis for the fishing nets sample with and without catalyst. In the case without catalyst, fishing nets has a minor peak around 2300 cm^{-1} referring to the presence of CO_2 [27], which was

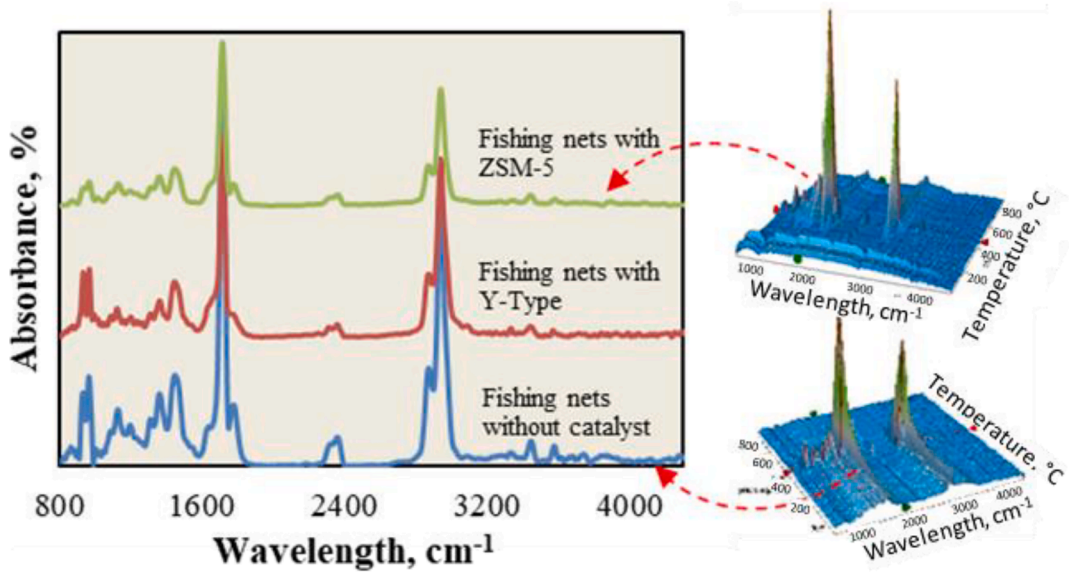

Fig. 5. The 3D-2D FTIR spectra analysis of the fishing nets with and without catalyst.

decrease by adding the catalyst. Moreover, some oscillations were observed around 1300 and 1500 cm^{-1} which indicates the presence of the N—O group [27], while valence carbonyl group oscillations around 1700 cm^{-1} indicate aldehydes and ketones presence. At 3600 cm^{-1} minor obertonic oscillations indicates amine (N—H) group from nylon-6 thermal decomposition.

A sharp peak in the fishing net sample was noticed around 3000 cm^{-1}, referring to C—H, which are typical for methane and aromatic-like compounds [28]. The absorbance of hydrocarbons (C—H) and aromatic compounds slightly increased because the non-stable hydrocarbons were combined together to form polyolefins. During this process, a higher number of flammable compounds and oil might be obtained [29,30]. This might be proven by the presence of visible peaks at the fingerprint zone of 600 – 1000 cm^{-1}, mainly attributed to aromatic compounds [31]. Moreover, previous studies shows, that the amount of flammable derivatives is directly equal to the heating rates [16], the catalyst did not significantly affect the indicated FTIR spectra peaks.

3.3. Pyrolysis experiments at the laboratory-scale bench

3.3.1. Temperature influence to the gaseous pyrolysis products generation

Gaseous products composition was measured using two methods, which were described in paragraph 2.4. For understanding the feedstock thermal decomposition during the pyrolysis process and gaseous products release by gradually increasement of process temperature, the semi-online gas analyzer VISIT 03H was used. Commonly, the gaseous products after the thermal treatment of fishery plastic wastes are composed of carbon monoxide (CO) and dioxide (CO_2), hydrogen (H_2), methane (CH_4), and other light hydrocarbons, such as C_2H_2, C_2H_4, C_2H_6, etc. [32]. The evolution of gas resulted in the process for the seaweed pyrolysis is depicted in Fig. 6.

The gas formation starts at around 300 °C and is continuous until the end of the test. As can be seen from the results, the thermal conversion process was processed under the complete absence of oxygen with the full presence of N_2 during the entire procedure (Fig. 6 a). This indicates that the conversion process of fishing nets was conducted in the perfect case under the typical conditions [33].

First of all, temperature influence for the pyrolysis products formation must be widely explained. All experiments were performed in a range of 500 – 900 °C, what shows very clear tendencies. As could be seen in the Fig. 6-a, the amount of the N_2 in gas composition starts to decrease significantly once it reached the end of the treatment (500, 700 and 900 °C), indicating the products formation. The increasement in temperature significantly affect the gaseous products generation: the amount of CO increased from 0.3 to 4.9 %, C_nH_m from 1.4 to 27.4 %, and H_2 from 0.3 to 4.6 %, while CO_2 was reduced from 2.3 to 0.1 %, when the temperature increased from 500 to 700 °C. Second increasement (from 700 to 900 °C) changed the composition all components significantly: CO decreased from 4.9 to 0.8 %, C_nH_m from 27.4 to 2.4 %, H_2 from 4.6 to 1.9 %, and CO_2 increased from 0.1 to 6.7 %. Different emission areas could be explained by the not homogenous fraction into the feedstock: PP and PE has a different thermal degradation and pyrolyze step by step, leading to appearance of gas components at different conspicuous peaks [21,34]. It was investigated that in the final pyrolysis stage, the gaseous products emission peaks became non-linear owing to the higher molecular mass polymeric chain decomposition (occasional scission of chains or at the end of chain) in the weakest parts of the bonds (sp3 link) [35], leading to the formation of monomers and radicals, which underwent into the condensation reactions and compose some gaseous phase products [36]. Moreover, the higher added value gaseous products (C_nH_m and H_2) were obtained after the temperature reaches 500 °C resulting of degradation of main polymer (nylon-6), and these observations agree with TGA-DTG results from the section 3.1. The optimum temperature for the valuable gaseous products generation was investigated to be around 700 °C. Nevertheless, based on these results it is clear, that temperature has a significant influence on the formed

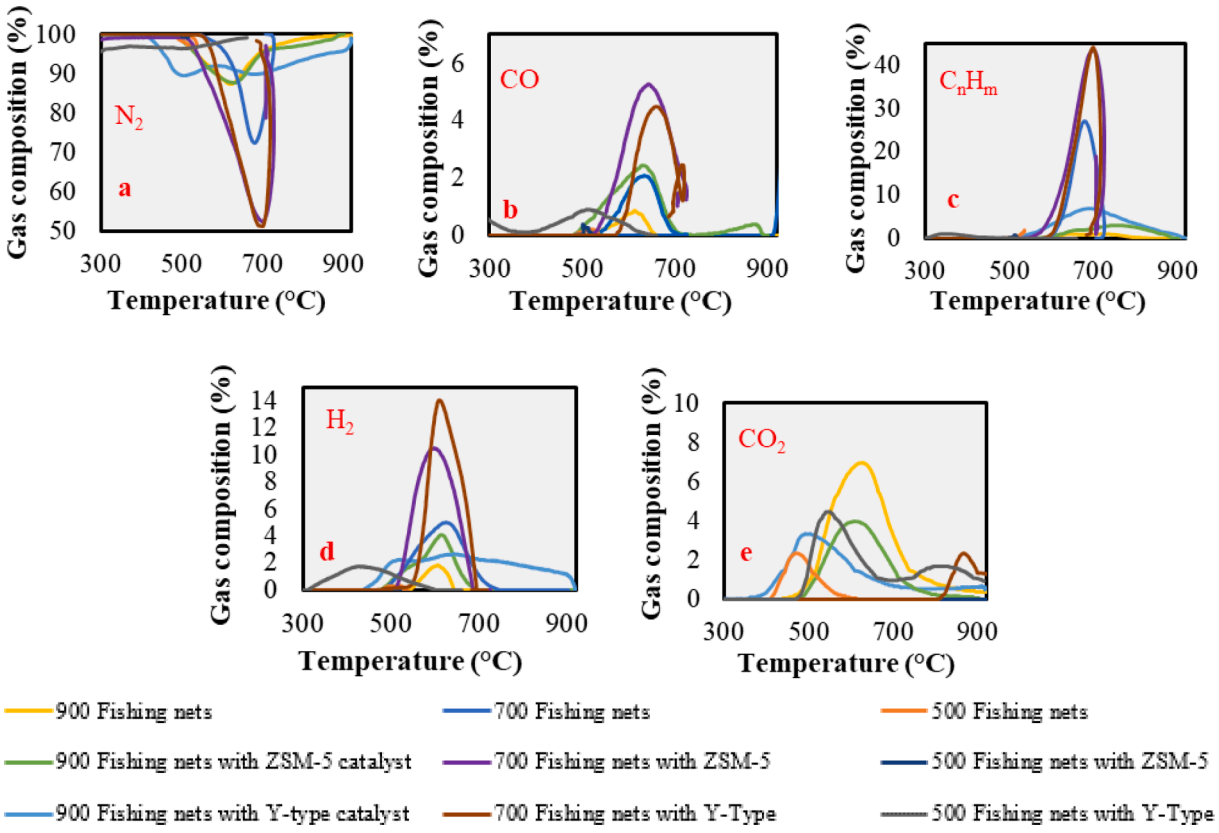

Fig. 6. The gaseous products analysis during the pyrolysis of the fishing nets.

pyrolysis gaseous products, especially for flammable gasses (C_nH_m and H_2), which shows the tendencies, that the amount of these derivatives had increased by the increasement of temperature, resulting of decomposition of remaining organic components from the residual fraction [37].

The catalyst influence for the pyrolysis process is significant and the results are presented in Fig. 6. As could be seen from the presented data, the amount flammable gaseous products were increased significantly by the both catalyst (ZSM-5 and Y-Type). Catalytical process forces the emission of C_nH_m derivatives and the amount increased from 27.4 to 43.5 and 43.4 % with ZSM-5 and Y-Type catalyst, respectively, while the amount of H_2 increased from 4.6 to 10.2 and 14.1 %. The yield of CO_2 was decreased by the catalyst because of CO_2 ability to react with H_2 under the 700 and 900 °C pyrolysis temperatures and formulate a C_nH_m molecules, thus resulting in a very high yield of these derivatives [38]. Obtained results correlate with literature review results, as occurred the same tendencies of polyamide plastic wastes decomposition using catalyst during the pyrolysis process [13,16].

It has to be mentioned that a continuous gas composition measurement device (VISIT 03H) for CH_4 measurement uses IR (infrared) detector (measurement cell). It means that about 3000 cm^{-1} wavelength captures C—H valence oscillations. Based on the literature, there is a high possibility to form other light hydrocarbons, such as C_2H_6, C_3H_8, etc., and it would be detected as a CH_4 compound, which would provide inaccurate analysis and measurements. Therefore, GC was used to investigate the reliability of the data obtained and to check the probability of finding other light hydrocarbons mixed with methane, as shown in the next paragraph.

3.3.2. *Temperature influence to gaseous pyrolysis products generation based on GC/Ms*

In order to investigate the precise chemical composition of the formed pyrolysis gases, the gas chromatograph with thermal conductive detector (GC/TCD) was used. A portable continuous flow sampler performed the analysis on six "Tedlar" bags filled with pyrolysis gases from the specified batches. The sampling was performed during the pyrolysis process based on the decomposition profiles determined by TGA results, particularly the major degradation zone before the end of the reaction.

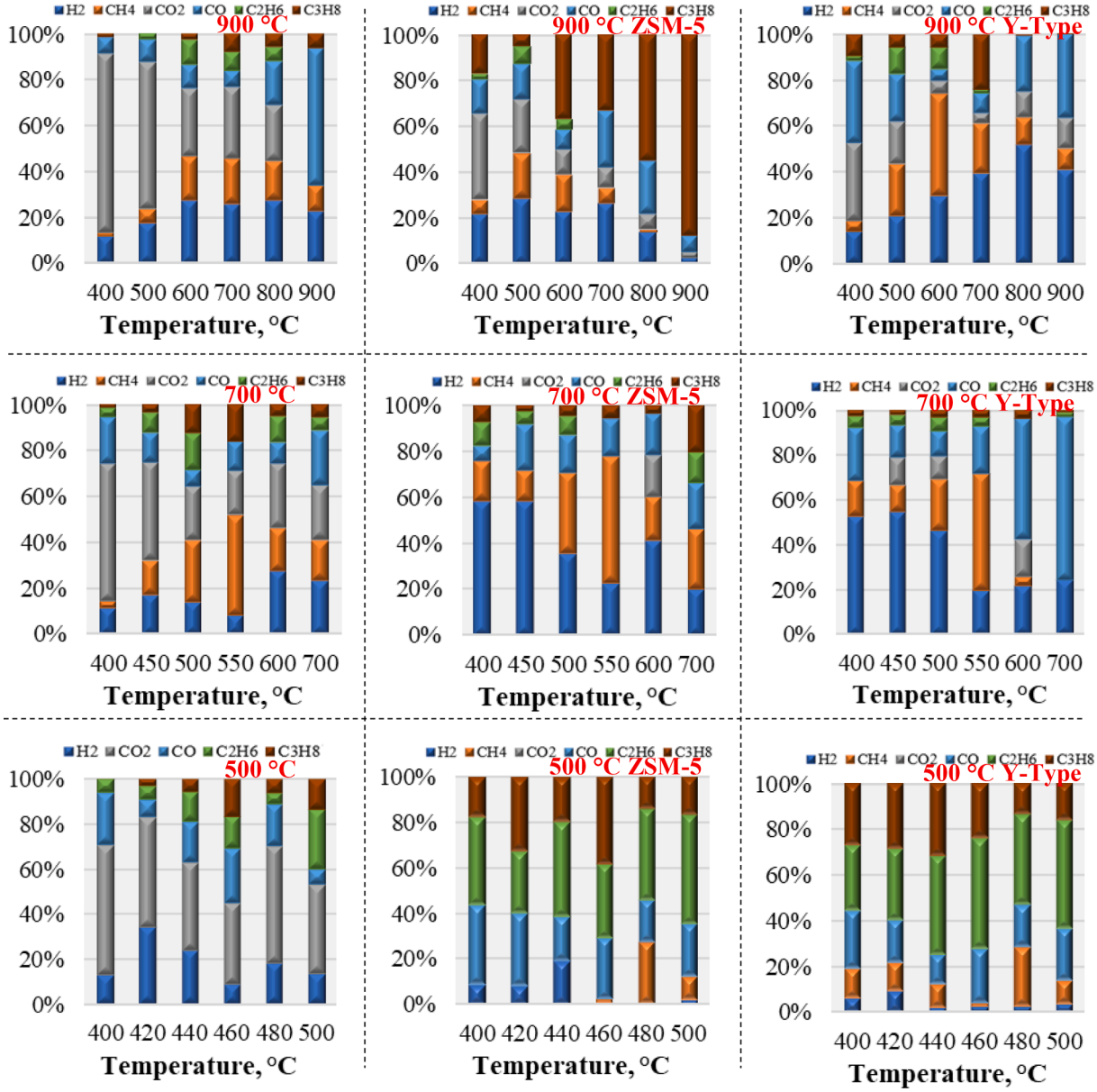

Fig. 7. GC/Ms analysis of the pyrolysis gases at specific temperatures.

In the Fig. 7 are depicted the results of the formed pyrolysis gases from the waste fishing nets with and without catalysts at the specific temperatures. The results are presented without balance N_2 gasses. As could be seen from the results, the highest temperature (900 °C) leads to the formation of CO and CO_2, with some H_2 and CH_4, while only the minor footprints of C_2H_6 and C_3H_8 was detected. Decreased temperature showed almost the similar tendencies as it was widely described in the previous section: the composition of C_nH_m and H_2 was increased at 700 °C and decreased at 500 °C. Based on the GC results it is clear that light hydrocarbons, such as C_2H_6 and C_3H_8 was mixed together with CH_4, giving discrepancies to the IR cell for online measurement device. The maximum peaks of these gaseous products reach 33, 11, and 13 % of CH_4, C_2H_6, and C_3H_8, respectively.

Pyrolysis at the lowest (500 °C) temperature increased the amount of C_3H_8 because of inability to decompose the bonds between the atoms of resulting not high-enough temperature. Moreover, the results show, that at the lower sampling temperatures the concentration of CO_2 and CO in all batches were high, while as the temperature increased, the concentration of flammable gasses increased, and these results agree with the results from online measurement device, and the literature [39].

Catalyst significantly increased the concentration of flammable gaseous products leading to the formation of H_2 and CH_4. The reactions under 700 °C showed the potential for hydrogen recovery, as the catalyst ability to decompose the higher molecular mass derivatives on the specific surface-active spots. ZSM-5 and Y-Type catalyst increased the amount of H_2 by the 34 and 35 %, while the amount of methane increased by 15 and 11 %, respectively. The amount of other light hydrocarbons slightly decreased by the catalytical thermal reaction mechanism. The reactions under 500 °C increased the amount of low molecular mass hydrocarbons by the H_2 ability to react with CO_2 forming C_2H_6 and C_3H_8 [38]. The amount of these compounds increased by the 14–12 %, and 21–23 %, with ZSM-5 and Y-Type catalyst, respectively.

3.3.3. *Analysis of the pyro-oil products*

In order to evaluate liquid fraction composition, a gas chromatograph with a mass spectrometer detector (GC/MS) has been used. All liquid products were divided into two parts, called the light fraction and the heavy fraction. The liquid products collected after the condensation at a low-temperature process are called as a light fraction or condensable gases, while the instantly collected pyro-oil products are as called heavy fraction or heavy tars. The chromatography measurement results are

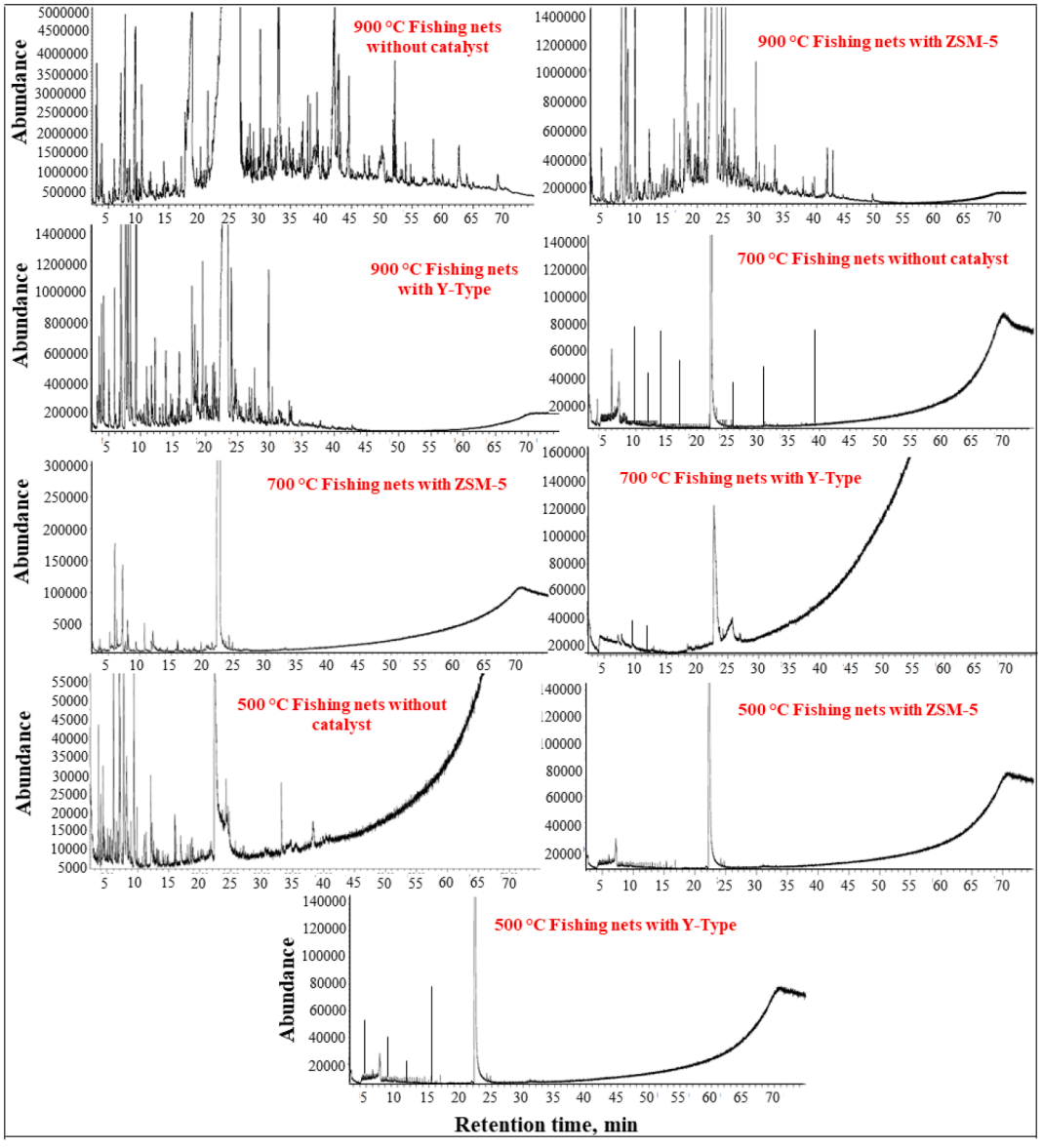

Fig 8. GC/MS spectra of the formulated light pyro-oils from the waste fishing nets sample.

presented in the sections below.

3.3.3.1. Light tars analysis. Fig. 8 shows an analysis by GC/MS of the fishing nets light tars with and without catalyst at the different temperatures after the thermal treatment. As could be seen from the results, the GC–MS chromatogram showed that both fishing nets and fishing nets-catalyst batches contained several hydrocarbon fractions with different variation in their composition and peak areas. The main compounds at 900 °C pyrolysis light tars are variously substituted phenolic compounds (21 %), some low molecular mass acid (11.2 %) and pyridine compounds (31.4 %). Moreover, the abundance of caprolactam reaches 9.4 %, which was obtained by the decomposition of main polymer (nylon-6) into the feedstock [40]. The similar tendencies were obtained with decomposition over both of catalyst. However, it was noticed that the abundance of some fractions increased after having included catalyst to the reactions, especially caprolactam, where abundance reaches 21.2 and 18.7 % with ZSM-5 and Y-Type catalyst, respectively. Meanwhile, higher molecular mass compounds, such as pyridine compounds, decreased significantly to 11.1 and 8.4 % due to its decomposition into acetic acid (8.4 and 11.3) and aliphatic hydrocarbons by the ring opening reactions promotion.

The presence of caprolactam and polycyclic phenolic compounds present into the condensable gaseous products batch indicates that the condensation process was not sufficient, therefore, it is highly recommended to use other techniques to separate these products, such as membrane processing or additional condensation [41,42].

The decreasement of the pyrolysis temperature showed, that the products distribution converted into the specific derivatives, such as toluene (17 and 13 %), p-xylene (16 and 14 %), 2,4-Dimethyl-1-heptene (15 and 12 %), and caprolactam (74 and 93 %) derivatives, at the both 700 °C and 500 °C temperatures, repetitively. Catalyst increased the amount of caprolactam polymers at both cases, leading to the decreasement of the other compounds.

It may be explained by the catalyst and temperature synergetic approach for the feedstock: the decomposition of the long polymeric branched nylon-6 chains to the monomeric polymer caprolactam chains at the specific C—C and C—N bonds was initiated by the catalyst active spots in the structure and not high-enough temperature to start the chaotic reactions [16,43]. Moreover, the temperature tend to promote the caprolactam thermal decomposition, leading to the formation of 5-

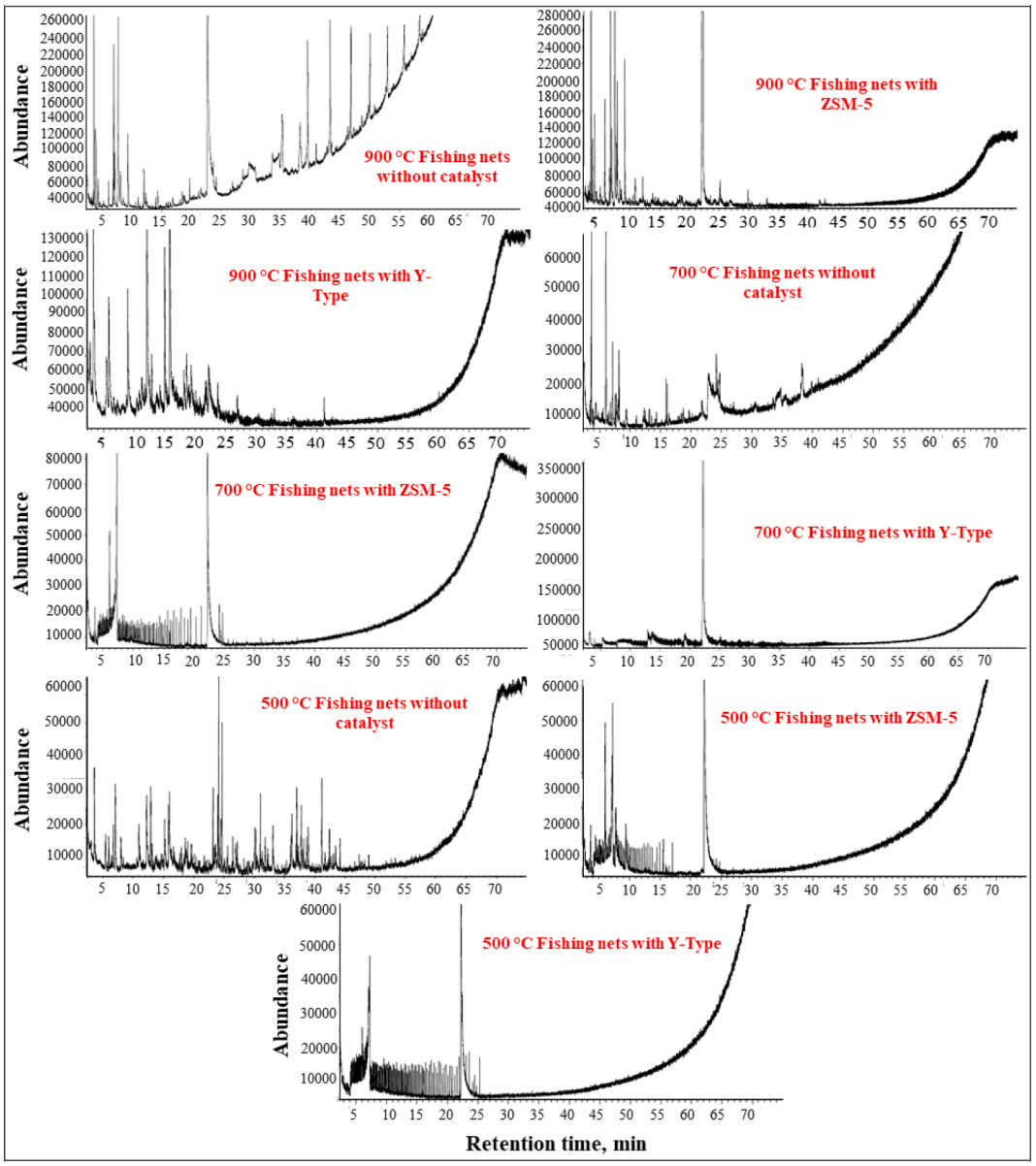

Fig. 9. GC/MS spectra of the formulated heavy pyro-oils from the waste fishing nets sample.

hexaneamide compounds, while this process was suspended by the catalyst working mechanism [44].

3.3.3.2. Heavy tars analysis. The heavy tars are the liquid products obtained during the pyrolysis with a typical condensation process, just without any chemical absorption. Fig. 9 shows the GC/MS spectra of the heavy tars after the fishing nets pyrolysis with and without catalyst at the different temperatures. As seen from the spectra, a high amount of high molecular mass tars was generated (in the case without catalyst). It was determined that the pyrolysis under 900 °C heavy liquid fraction composition is mainly composed of polysubstituted benzene derivatives, such as toluene, ethylbenzene, o-xylene, naphtalene, and variously substituted phenolic compounds. Moreover, the catalyst decreased the amount of higher molecular mass derivatives, converting them into the light hydrocarbons [45].

The amount of caprolactam was also decreased because of the decomposition reactions and even light compound formation. Interesting tendencies was observed with synergy of Y-Type catalyst and high temperature, where the biggest part of caprolactam was dismembered into the lighter molecular mass 5-hexaneamide compound by the earlier mentioned mechanism [44]. The temperature decreasement showed interesting trends for caprolactam recovery: more than 80 % of this compound could be recovered using 500 (83 %) and 700 °C (96 %) temperature with Y-Type catalyst. This observation could be explained by the mechanism from the previous paragraph, based on the not-high enough temperature to create the chaotic decomposition reactions, leading for depolymerization process and monomers recovery [16]. Other tendencies were the similar as it was with light tars analysis:

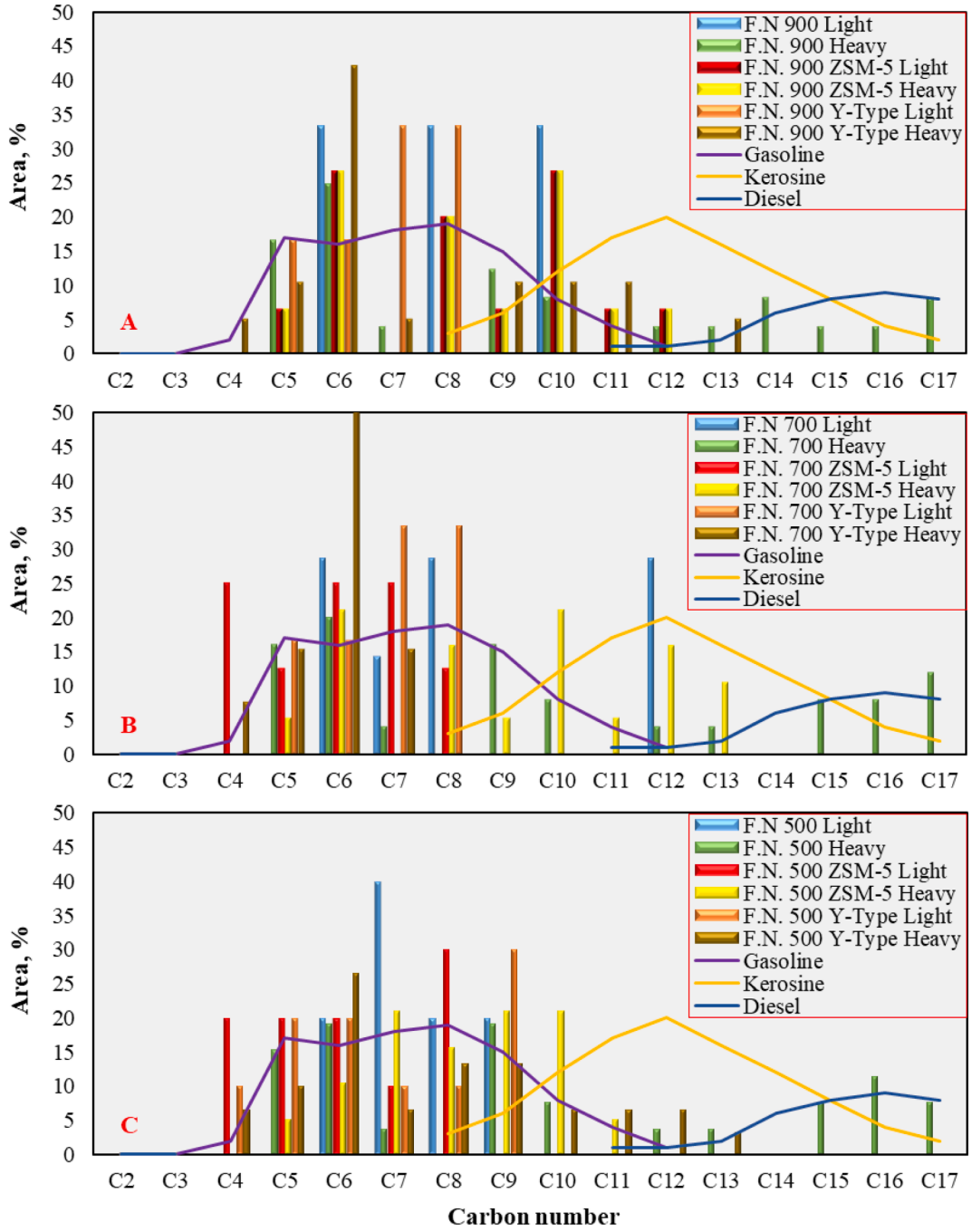

Fig. 10. Liquid products distribution is based on the carbon number. A – 900 °C, B – 700 °C, C – 500 °C.

catalyst decreased the amount of heavier molecular mass compounds by the decreasement of temperature with additional amount of catalyst.

In general, analysis of the formed tars clearly shows the possibility of the additional valuable chemical's recovery from the waste's feedstock. Moreover, caprolactam recovery from the waste fishing nets could be assigned to added value product recovery and contribute to creating a circular economy. These tendencies show alternative pyro-oil's uses obtaining high-quality hydrocarbons and caprolactam monomers, which could be recovered and used in nylon-6 synthesis. In order to investigate the possible benefits of a received pyro-oil product as an alternative fuel from wastes, the distribution based on carbon atoms was described in the next paragraph.

3.4. Fractional composition of wastes pyro-oil

The evaluation of liquid product composition and distribution based on the carbon atoms was maintained based on the GC/MS results described in the previous paragraph. The obtained results were evaluated as a possible alternative fuel from wastes compared to gasoline, kerosene, and diesel fractions, and the data are presented in Fig. 10. The atoms of carbon distribution in bio-oils from the different feedstocks are in the range of C4 to C17. As seen from the presented results, both types of catalyst highly decreased the number of carbons compared to fractions without catalysts. The fraction from C12 to C17 was decomposed and converted into the most abundant fraction (C4-C9). The working catalyst mechanism could explain it based on the ring-opening reactions and higher molecule's dissociation into the lighter aliphatic carbon chains. Moreover, the catalyst decreased the amount of N- and O-heterocyclic compounds, increasing bio-oil quality [46].

Decreasement of the temperature showed, that the distribution of fractions was more equal to each other in comparison with the higher temperature results. This may lead to the optimum condition's determination for the specific fraction formation, recovering only the selected polymeric length chains [47].

In general, this analysis shows that the catalytical thermal treatment of the marine plastic wastes is a possible solution for the refuse's utilization and higher quality pyro-oil generation. Moreover, formed pyrolysis tars after the catalytical treatment corresponds to the gasoline fraction (based on the number of C atoms analysis) and could be used in further investigations. It is known that some types of heteroatoms are also obtained into these products, so for wider uses, additional treatments, such as hydrocracking, must be adjusted.

3.5. Conversion efficiency during the pyrolysis process

The conversion efficiencies have been calculated to evaluate the product yields and catalyst influence on the product formation. A mass balance was considered for pyrolysis products: it was determined by primary feedstock mass and terminal mass of formed pyrolysis oil, pyrogas, and biochar. Also, the conversion of the selected feedstock to liquid and gaseous product percentage was estimated. The results are presented in Table 2.

The tendencies of the formed products are typical for the thermal decomposition process: diminution of the temperature leads to the increased formation of the liquid's products, and decreased gaseous products emission. The highest amount of liquid products was obtained at 500 °C with ZSM-5 catalyst, while the highest amount of gaseous products recovered at 900 °C without catalyst. It could be explained by the catalyst working mechanism in the pyrolysis process: Y-Type catalyst restraining the formation of aldehydes, acids, and ethers, promoting the formation of the light aromatic derivatives, while ZSM-5 catalyst increases the amounts of aromatic derivatives, affecting the amount of oxygenates towards deoxygenation reactions [48]. Nevertheless, based on the chemical products analysis (compartment 3.3) could be stated, that pyrolysis at 700 C with ZSM-5 catalyst is optimum conditions for the higher added value energy products recovery, because of the highest amount of hydrocarbons into the gaseous phase, and the highest purity liquid products recovery [49]. Obtained results correlates with other author results of plastic's pyrolysis, where the scientists investigated that for the higher amount of gaseous and char products recovery, higher than 550 °C is suggested, while the preferred liquids products recovery should be maintained at the lower temperatures [35,50].

Table 2
Pyrolysis product yields.

Sample	Pyrolysis oil, wt.%	Pyrolysis gas, wt.%	Pyrolysis char, wt.%
Fishing nets 900 °C	67.63	32.02	0.35
Fishing nets Y-Type 900 °C	69.63	29.98	0.39
Fishing nets ZSM-5 900 °C	70.15	29.24	0.61
Fishing nets 700 °C	71.21	28.38	0.41
Fishing nets Y-Type 700 °C	73.22	26.30	0.48
Fishing nets ZSM-5 700 °C	74.68	24.69	0.63
Fishing nets 500 °C	73.11	26.58	0.31
Fishing nets Y-Type 500 °C	76.41	23.17	0.42
Fishing nets ZSM-5 500 °C	77.22	22.23	0.55

4. Conclusions

In this research work, the catalytic pyrolysis of waste fishing nets over Y-Type and ZSM-5 catalysts under different temperatures (900-700-500 °C) was studied.

First of all, the primary characterization of selected feedstocks was maintained using the TGA-DTG-FTIR system. The waste fishing nets have a significant peak around 440 °C with a total mass loss of 88 wt%, which could be assigned to main polymer nylon-6 thermal degradation. The most relevant functional groups in gaseous products are C—H, C═O, O—H, and N—H, which represent aliphatic and aromatic hydrocarbons, aldehydes and ketones, alcohols, and amines present.

The gaseous, liquid and solid fractions were obtained during the pyrolysis at a laboratory-scale bench. It was noted that the most relevant compounds in gaseous products are CO, CO_2, CnH_m, and H_2. Variations between the temperatures showed, that the highest amount of valuable gaseous products were obtained at 700 °C, as 27.4 % of C_nH_m, and 4.6 of H_2. Catalyst significantly increased the amount of these compounds, and the optimum conditions were set to be around 700 °C over Y-Type catalyst, which increased the amount of C_nH_m up to 43.4 % and H_2 up to 14.1 %.

Liquid products analysis showed promising results for caprolactam recovery: catalyst and temperature variations showed that more than 80 % of caprolactam may be obtained in the batch with Y-Type catalyst at 500 °C (83 %), and 700 °C (96 %). Together with this polymeric monomer recovery could be also regained some energy products, leading to the optimization of technology for both of these valuable higher added value derivatives recoveries.

The mass balance calculation showed accurate tendencies, that decreasement of the temperature increased the formation of the liquid products up to 73.1 %, while the amount of the gaseous products decreased to 26.6 %. Catalyst slightly increased the amount of liquid products at the maximum value of 77.2 %, significantly affecting the composition of the formed derivatives.

This study shows that the pyrolysis process is suitable for waste fishing nets utilization, and obtaining additional energy products. Moreover, high caprolactam content could be recovered from the fishing net sample as an additional higher-value product, which could be reused to synthesize nylon-6. Therefore, this material showed potential as feedstock for higher added value energy products production,

contributing to the creation of a circular economy.

CRediT authorship contribution statement

J. Eimontas: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Resources, Software, Supervision, Writing – original draft, Writing – review & editing. **N. Striūgas:** Conceptualization, Data curation, Formal analysis, Investigation. **K. Zakarauskas:** Data curation, Investigation, Methodology, Formal analysis. **I. Kiminaitė:** Data curation, Investigation, Methodology, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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