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ABSTRACT AND SCOPE

The MARine Litter cross-border awarenESS and innovation actions (MARLESS) aimed to address the problem of marine litter in the Adriatic Sea from multiple fronts, to achieve visible and concrete results. In fact, there are numerous factors that intervene in the creation of Marine Litter, the plastics coming from the inside, from tourism or the aquaculture sector.

The project's approach was therefore to address the problem with a holistic approach, jointly intervening on the scientific and monitoring aspects.

As a treatment process for plastic waste collected at sea, it was decided to use pyrolysis, which allows, in addition to a chemical recovery of the plastic components, also the recovery of energy.

One of the advantages of the pyrolysis process is that all the products have commercial value: the gas can be recycled to feed the process heat, the oil can be refined, if necessary, to produce chemicals and fuel, and the solid product can have many applications.

The output of the MARLESS project is a procedure for the treatment of marine plastic waste, adapted to the different types of plastic collected, which can be implemented both on land and at the sea using a boat dedicated to the collection and treatment of plastic waste at sea.

In this deliverable, the positive and negative aspects of each phase carried out in WP 6.2 will be considered, including improvements to make, and comparing the data from the project with those present in the literature.

1. INTRODUCTION

Among the several methods and processes for recycling plastic waste, pyrolysis, a thermochemical process, was used in WP 6.2. It is part of the chemical recycling processes, which consists in the depolymerization of the plastic into smaller molecules or monomers (Lech, 2020).

Pyrolysis is a process that takes place at temperatures greater than 300 °C in the absence of oxygen and produces solids, gases (e.g. CO, CH₄ O H₂) and an oil that can be introduced directly in petrochemical transformation chains or other synthesis processes and transformed into useful chemicals (INAcclimate, 2020) (Crippa, 2019).

Pyrolysis makes possible the treatment of a wide range of plastic raw materials, such as polyethylene, polyethylene terephthalate, polystyrene, etc. Therefore, starting from a very inhomogeneous base material, a wide range of recoverable chemical components is made available, with appropriate treatments of the pyrolysis products (INAcclimate, 2020) (UMSICHT, 2020).

Overall, the chemical recycling processes reduce the landfilling of plastics, and promote their energy use. Furthermore, the basic technical feasibility of chemical recycling processes was demonstrated thanks to the presence of functioning pilot plants (Hofmann, 2021).

2. CHARACTERISATION OF THE INPUT

For the pyrolysis process tested in the MARLESS project, the plastic was subjected to 3 physical pre-treatments: sorting by polymer (other non-plastic waste was not considered), drying and shredding.

2.1. Sorting

The sorting was manual and visual, so errors may be made regarding the identification of the type of plastic. Furthermore, it may be influenced by the site of collection.

The floating plastics, collected along the beach and from the fishermen's nets, were almost intact, with a visible plastic recycling code, which identify the type of material of which an object is made. When fragments are present, these were all gathered in a single set of unidentified mixed plastics.

On the contrary, all plastics collected from the seabed were dirty, wet and slightly degraded. Therefore, recycling codes were not distinguishable; the separation by polymers was carried out according to the most common sector of application for a given polymer:

- PS (polystyrene): yogurt jars, disposable cups and cutlery, coat hangers, egg wrappers, plastic models, plates, tubs, straws, mussel nets;
- PE (polyethylene): detergent containers, bottles for containing detergents or food, "bubble film" (or bubble wrap), bags, food films, coating film for electric and telephone cables, plastic caps;
- PP (polypropylene): packaging, DVD cases, sheets and pipes for the building industry;
- PET (polyethylene terephthalate): containers for liquids, bottles of shampoo and soaps, trays for the fridge and oven, containers for food, bottles of water and soft drinks;
- PVC (polyvinyl chloride): imitation leather, wire coatings, credit cards;
- PU-PS (polyurethane and polystyrene): expanded polyurethane foam is injected into the cavity walls to improve thermal insulation.

Excluding the fraction of non-plastic wastes (water, mud and sand), the mass distribution of the identified plastic polymers, was the following (a range is given, depending on the different samples):

- PS: 0.3% e 0.7%-4.9%
- PE: 10.5% e 1.4%-11.9%
- Nets in PP: 56.5% e 2.3%-19%
- PP: 0% e 0.5%-2.7%
- PET: 3.4% e 3.4%-18.9%
- PVC: 0.3% e 0-0.6%
- PU-PS: 0% e 0.1-0.5%
- Mixed plastic: 0% e 5.9%-15.1%
- No-plastic waste: 15% e 19%-51%
- Scrap (water, sand, mud): 13% e 9%-34%

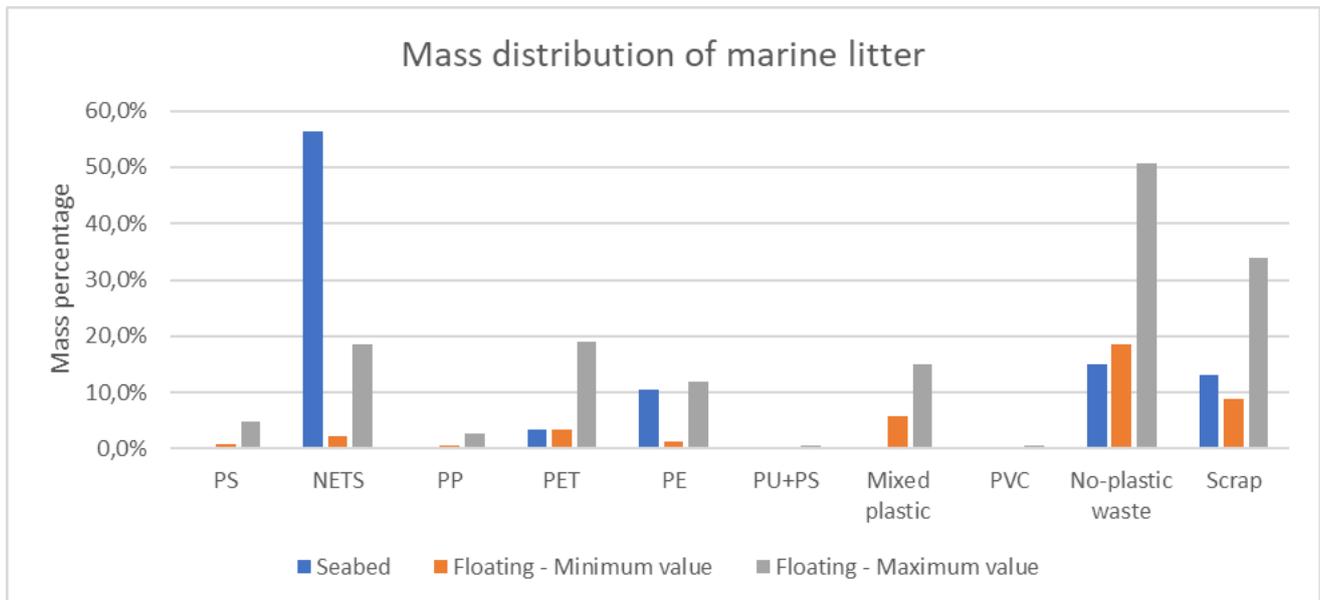


Figure 2.1 Percent mass distribution of all marine litter

In the work of Hee and Schlögel (2021) (which we will discuss in session 3), a sorting of beached plastics was carried out, collected in 2 different areas, with the following percentages by weight:

- Plastic 3D: 88% (PET, PP, HDPE, PS, PUR) e 15% (PS, PP, PVC, HDPE, ABS)
- Films: 10% (ABS, PP, PVC, HDPE, PS) e 6% (PA, PP, PVC, HDPE, PS)
- Nets: up to 55%
- Rubbers and elastomers: up to 8%
- Foamy plastic: <<1% e 5% (PET, PS, HDPE, PUR)
- Others: 1% e 9%
- Metals: 1% e 2%

2.2. Final Consideration

The plastic collected from the seabed was dried before treatment, while with the beached ones, only the PET fraction was found to be too humid for pyrolysis. However, if the residual humidity value of the whole set of beached plastics collected, not separated by polymer, is considered, then the value falls within the parameters required by the pre-treatment for pyrolysis. So, the beached plastics, mixed together, can be directly pyrolysed.

During the pyrolysis phase, laboratory-scale experiments were carried out on individual plastic fractions. Among the samples tested, two pairs were identified to be made by the same polymer: BAGS and PE of polyethylene, and NETS and PP of polypropylene.

GC-MS analyses were carried out for all the samples, thanks to which the content of aliphatic and aromatic compounds of pyrolysis oils were determined.

These analyses demonstrated that the manual sorting was very accurate, free of significant errors, as the results obtained for two samples of different objects (subject to error in identifying the polymeric class they belong to) such as PE and PP, were comparable to the results obtained from samples of BAGS and NETS, containing the same polymer.

The results obtained, from the pyrolysis of the single plastic fractions, showed that the sorting method, used in this project, was very accurate. Therefore, it is possible to replicate it, without resorting to specific equipment for the identification of plastics.

3. PLASTIC MARINE LITTER PYROLYSIS

Many studies were conducted on the pyrolysis of plastics, however the number of studies dealing exclusively with the pyrolysis of marine plastics is limited, compared to the pyrolysis of plastics collected from municipal waste. Furthermore, in many studies calcium oxide was used as an additive to avoid corrosion problems in the reactor, or catalysts were used for the pyrolytic process. As a comparison with the work done for MARLESS, the results obtained from two studies carried out on the pyrolysis of marine plastics are reported:

- Hee and Schlögel (2021): By thermo-gravimetric analysis, the processes of chemical recycling (pyrolysis, gasification) and energy recovery (incineration) of marine litter, consisting of two heterogeneous marine litter samples, were reproduced. In addition, semi-technical scale pyrolysis trials were conducted, and residues were analyzed by proximate, ultimate and X-ray fluorescence analysis. Only pyrolysis-related results were considered.
- Veksha and Ahamed (2022): thermochemical recycling of plastics, first subjected to pyrolysis, then to chemical vapor deposition. Again, only results relate to pyrolysis were considered.

In both studies, no catalysts or additives were used, but only the pyrolysis of a single mixed plastic sample was carried out, while in MARLESS the pyrolysis of the different polymeric fractions was also carried out. Then, results of two studies were compared with only results of the batch-scale pyrolysis experiment of a single mixed plastic sample.

3.1. Pyrolysis reactor

In the MARLESS project, the pyrolysis of mixed plastics was conducted in a batch-scale rotary kiln. Before the start of the experiment the reactor was filled with the feedstock (marine litter) and afterwards flushed with nitrogen (1L/min) to create an inert atmosphere. The reaction chamber (drum) was set in rotation around the horizontal axis at a rotational speed of approx. 5 revolutions per minute. This ensures that the material remains in motion during treatment and is heated equally from all sides. The reaction chamber was heated to the target temperature of 600 °C using an electric heater jacket. Reaching the set temperature took approx. 15 minutes. Upon reaching the target temperature, the treatment period of 30 minutes started. Emerging gases and vapours flowed from the reaction chamber into a condensation unit (shell and tube heat exchanger), where the vapours were condensed into the liquid product. After the condensation unit, a gas extraction station was installed using a bypass. At this point, the gas composition was determined during the experiment. The remaining gas was released into the environment after passing an activated carbon filter. After the treatment period expired, the heater was turned off while the reaction chamber continued to rotate. As soon as the temperature in the reaction dropped below 200 °C, the rotation was also stopped.

The solid product was collected in the reaction chamber, and the liquid product was collected in the condensation unit and in the collection container mounted to it. The mass of gaseous product was determined as the difference between the mass of treated material and the masses of solid and liquid product and loss (small quantities of products that cannot be transferred to the collection containers and weighed, estimated at 2% of weight of input material).

Subsequently, pyrolysis products were subjected to a CHNS analysis and an X-ray fluorescence (XRF) analysis, their calorific values were calculated and, by GC-MS, the composition of the liquid product was determined.

In the analysis by Hee and Schlögel (2021), pyrolysis trials were conducted in a modified chamber furnace containing a stainless-steel retort as a fixed bed reactor. To ensure oxygen exclusion and thus pyrolytic conditions, the retort was continuously flushed with nitrogen. The furnace was heated up at a rate of ca. 8–10 K/min. To minimise the risk of sudden pressure rises the heating program involved a 30 minutes plateau at 250 °C before heating up to the target temperature of 700 °C and holding for another 30 minutes.

The pyrolysis gas produced in the retort was led through steel containers tempered to –20 °C to collect all condensable components. The remaining permanent gas fraction was burned in a post-combustion chamber to minimise harmful gas emissions. After each trial, char and condensable product fractions were collected from the retort and the condenser respectively.

Subsequently, pyrolysis products were subjected to an elemental analysis and an X-ray fluorescence (XRF) analysis.

In the work of Veksha and Ahamed (2022), coastal plastic samples collected along a beach and underwater plastic samples collected by divers were used. From each sample, PET bottles were manually eliminated, and the remaining plastics (mixed together) were dried at 60 °C and shredded to <4mm. Also considering the influence of sea salt and biomass impurities on the thermochemical recycling, a part of both samples was washed with deionized water before being subjected to the thermochemical processing. But only unwashed sample results are considered in this report.

The pyrolysis was carried out in a vertical quartz reactor heated by an electric furnace. The reactor was purged for 10 min with 200 mL/min N₂ and heated at a rate of 10 °C/min to 600 °C in a 30 mL/min N₂ flow. The liquid products were collected in two condensers with the first condenser maintained at ambient temperature (23 °C) and the second condenser at 0–5 °C. The non-condensable pyrolysis gas was collected in a gas bag.

Ash content and calorific value of the pyrolysis products were calculated, and the composition of the liquid products was analysed by GC-MS, while the composition of the pyrolysis gases was determined by a gas chromatograph with a flame ionization and two thermal conductivity detectors.

3.2. Characterization of the starting material and yields of pyrolysis

In the MARLESS project, the mixed plastic sample (MIXED FRACTION), already shredded, consisted of the following percentages of polymer:

- PP: 48.8%
- PET: 10.6%
- PS: 4.6%
- MIX (plastic for which it wasn't possible to identify the polymeric class): 14.4%
- PVC: 0.9%
- PU-PS: 0.6%
- PE: 19.9%

Water content, ignition loss and calorific value were calculated for this sample:

- Water content: 0.50%
- Ignition loss: 96.34%
- Calorific value: 35.62 MJ/kg

CHNS analyses and XRF analyses were also performed on the sample. For the CHNS analysis, results in percent weight were:

- C: 71.97%
- H: 9.88%
- N: 0.34%
- S: 0.04%

For the XRF analysis, results in percent weight were:

- CHNS: 93.9%
- Ca: 1.74%
- Si: 1.7%
- Cl: 0.74%
- Al: 0.72%
- Fe: 0.51%
- K: 0.23%
- Ti: 0.12%
- S: 0.08%
- P: 0.07%
- Mg: 0.07%
- Br: 0.03%
- Zn: 0.02%
- Mn: 0.01%

After pyrolysis, product yields were: 11.2 % of solid product, 63.8 % of oil product e 23% of gaseous product.

Results, relating to the MIXED FRACTION, are similar to those present in the literature, except for the results obtained for the residual humidity and the oil yield, which depends both on the composition of the raw material and on the pyrolysis conditions.

In the analysis by Hee and Schlögel (2021), plastic samples were shredded, separated from the metal fractions and grinded to < 1 mm. Analyses were performed to define their proximal and elemental composition, with the following results in percentage weight:

- ash content: 10.55-13.52%
- volatile material: 84.6-91%
- C and H content: 66.8-68.2% e 9.2-9.5%, respectively
- N, S, Cl content: up to 0.7%, up to 0.6%, 0.5-1.3%, respectively

XRF analyses were also performed on the plastic samples before pyrolysis, with the following results, in percentage weight:

- Na: 0.7-2.2%

- Mg: 0.3-0.7%
- Al: 0.2-0.5%
- Si: 1.9%
- K: 0.1-0.3%
- Ca: 1-1.3%
- Ti: 0.3%
- Fe: 0.2-0.8%
- Zn: <0.2%

Furthermore, the calorific value of the plastic samples ranged from 33.43 to 35.97 MJ/kg. After pyrolysis, product yields were: 14.8-18.2% char and 21.5-24.1% oil.

In the work of Veksha and Ahamed (2022), after the removal of PET, beached and underwater plastics, before pyrolysis, had the following characteristics:

- residual humidity: 10.5% and 14.3% respectively
- marine plastic litter higher heating value: 36 MJ/kg and 30 MJ/kg respectively

After PET removal and drying, only 56.3% by weight of the beached plastics and 24.3% of underwater plastics were available for pyrolysis.

After pyrolysis, weight yield of oil, non-condensable gas and solid residues of pyrolysis were, respectively, 32.5%, 21.5% and 2.3% for beached plastic, and 11.9%, 9.4% and 3.1% for underwater plastic, with respect to the input plastics weight.

3.3. Solid pyrolysis residue

In the MARLESS project, for the SOLID FRACTION (solid product of pyrolysis), the water content, the loss of ignition and the calorific value were calculated:

- Water content: 2.92%
- Loss of ignition: 67.16%
- Calorific value: 26.37 MJ/kg

CHNS analyses and XRF analyses were also performed on the SOLID FRACTION. For the CHNS analysis, results in percent weight were:

- C: 59.40%
- H: 6.75%
- N: 0.86%
- S: 0.15%

For the XRF analysis, results in percent weight were:

- CHNS: 94%
- Ca: 2.37%
- Si: 1.32%
- Cl: 0.39%
- Al: 0.43%
- Fe: 0.50%

- K: 0.26%
- Ti: 0.30%
- S: 0.10%
- P: 0.07%
- Mg: 0.05%
- Br: 0.02%
- Zn: 0.12%
- Mn: 0.01%

These results are quite different from those present in the literature.

In the analysis by Hee and Schlögel (2021), the char samples, in the final analysis, had the following percentage weight: 20.3-49.7% C, 0.2-1% H, 0.3-0.7% N, 0.2 -2% S and 3-4.4% Cl.

The calorific value calculated for the pyrolysis chars obtained was between 8.59 and 18.44 MJ/Kg and XRF analyses of the same char samples showed a weight content of the following elements: 8% Si and Ca, 2-6% Na.

By comparing the compositions of the other pyrolysis products, it was observed that most of elements were concentrated in the pyrolysis char: 2% Mg, 1-2% Al, <1% K, 2-3% Ti, 2-4% Fe, <2% Zn.

3.4. Pyrolysis oil

In the MARLESS project, CHNS, XRF and GC-MS analyses were performed for the OIL FRACTION (liquid product of pyrolysis).

For the CHNS analysis, results in percent weight were:

- C: 83.52%
- H: 12.84%
- N: 0.11%
- S: 0.20%

For XRF analyses, results in percent weight were:

- CHNS: 99.1%
- Ca: 0.02%
- Si: 0.05%
- Cl: 0.29%
- S: 0.47%
- P: 0.04%
- Br: 0.02%

From the GC-MS analyses, it was observed that main components of the OIL PRODUCT were 2,4-dimethyl-1-heptene (33.66%) and styrene (18.62%).

Various aliphatic compounds with carbon numbers ranging from C8-24, and some other aromatics were also identified. Furthermore, the calorific value of the OIL PRODUCT was about 43.39 MJ/kg.

The results obtained, related to the calorific value and the CHNS analyses, are like those present in the literature.

In the analysis by Hee and Schlögel (2021), the oil samples, in the final analysis, had the following weight percentages: 72.7-77% C, 10.3-11% H, up to 0.4% N, up to at 0.1% S and 0.2-0.9% Cl. The calorific value calculated for the pyrolysis oils obtained was between 37.21 and 41.50 MJ/Kg; unfortunately, when looking at the ratio of similar iron, chromium and nickel contents between different samples during XRF analyses, it was seen that the analyzed samples had undergone cross-contamination with stainless steel during pre-treatment.

In the work of Veksha and Ahamed (2022), the pyrolysis oil was characterized by GC-MS. The analysis of the chromatograms of the two different oils obtained showed differences in the identified peaks, suggesting a different composition of the raw materials in the pyrolysed samples. For example, a styrene content of 3 mg/g was identified for coastal plastics, while for underwater plastics the styrene content was 29 mg/g, typical of PS pyrolysis (Park *et al.*, 2003). Furthermore, by grouping the carbonyl groups of oil into 3 groups, whose composition is similar to gasoline, diesel and heavy oil/wax products (Blazso, 2006; Ratnasari *et al.*, 2017), it was observed that in both oils 51% of the content is similar to gasoline range hydrocarbons (C6-C12), and that coastal plastics produce higher quantities of diesel, while underwater plastics produce higher quantities of heavy hydrocarbons.

3.5. Pyrolysis gas

In the MARLESS project, for the GASEUOS PRODUCT (gas product of pyrolysis), the gases CO, CO₂, CH₄, C_nH_m, H₂, O₂ and N₂ were measured every 20 seconds during the experiment, and the lower calorific value was also calculated, equal to 19.51 MJ/m³.

Average percentage values of each gas analysed, were:

- CO: 7.55%
- CO₂: 7.75%
- CH₄: 13.30%
- C_nH_m: 14.99%
- H₂: 1.16%
- O₂: 6.21%
- N₂: 49.04%

In the work of Veksha and Ahamed (2022), gases were characterized by GC with a flame ionization and two thermal conductivity detectors. While the amount of methane was the same for both samples, those from beached plastics had higher amounts of C1-C5 hydrocarbons than those from underwater plastics; this was attributed to the different composition of the plastic waste in the two samples.

3.6. Final consideration

The results of Hee and Schlögel (2021) showed that thermochemical recycling of marine plastics is possible. Pyrolysis only needs physical pre-treatments such as sorting and grinding, to remove part of the impurities, and the produced pyrolysis gas supplies the necessary process heat.

Furthermore, it was seen the output of gasoline-like oil fraction (C6-C12 hydrocarbons), obtained from the work of Veksha and Ahamed (2022), was less susceptible to the fluctuations of feedstock composition compared to other oil fractions, making marine plastics attractive for recovery and use as a fuel.

In the results obtained from the MARLESS project, it was observed that, for the pyrolysis process used, neither additives nor catalysts were required, and the presence of a carbon filter, in the gas line, prevented the dispersion of any polluting gases, such as HCl.

Calorific values of pyrolysis oil were found to be comparable to those of common fuels; however, the presence of chlorine and the high viscosity prevent its use as an energy source.

4. CONCLUSIONS

Considering the enormous variety of plastics and their dispersion in the sea, the pyrolysis of the individual plastic fractions divided by polymer does not seem feasible, due to the time required to collect enough mass of the same polymer, to reach the flow rate of the pyrolyzer at batch scale. On the contrary, the study conducted demonstrated the potential for pyrolysis of a mixed plastic sample. Further experiments need to be conducted, varying the composition of the plastic mix, to determine if and to what extent it affects the potential of pyrolysis oil as an energy source.

In addition to this, two other problems were highlighted.

Plastics collected at sea must always be dried and, for this, a certain amount of energy must be spent. Considering this problem, a system was devised, at a principal level, with which to dry the plastics, directly on the boat (Figure 4):

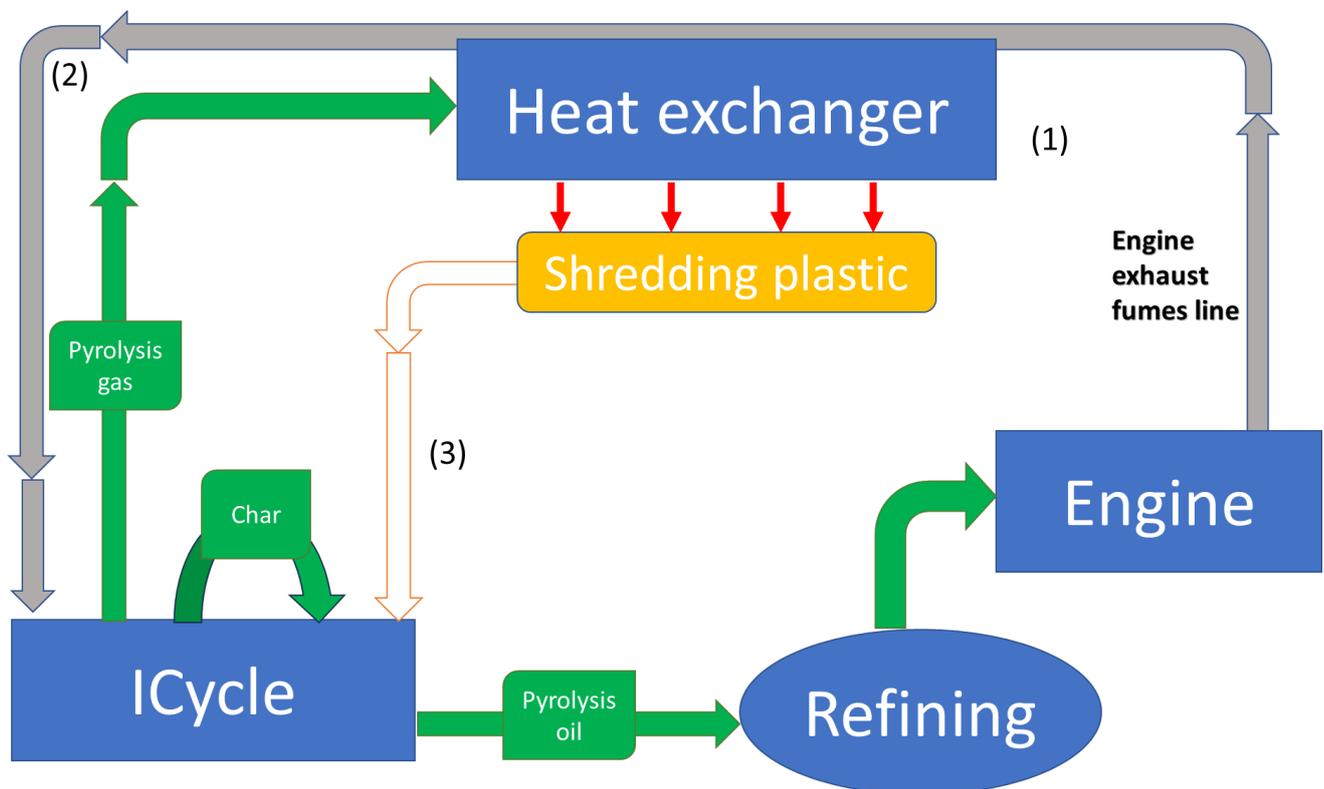


Figure 4 Ideal closed-circuit system for the self-sustainability of ships dedicated to the collection of plastics at the sea.

- While the plastics are removed from the nets, they are sorted, placing each polymer in each container, on the bottom of which there is a grate on which the plastics will rest. As the quantity of a given polymer increases, the underlying plastics are crushed, and lose the excess surface water, which will accumulate on the bottom of the container, under the grate.

- After a few hours, the plastics are shredded and dried, thanks to a heat exchanger (1), which draws heat from the engine exhaust line. Once the exhaust is sufficiently cooled, it is conveyed to the pyrolyzer (2);
- The dried plastics are then sent to the pyrolyzer (3)

By exploiting the heat generated by the engine exhaust, it is possible to dry the plastics, without excessive energy expenditure or the use of additional equipment, simply by conveying the exhaust gases towards the pyrolyzer, and on the way to it, placing a heat exchanger, which will lower the temperature of the fumes to pyrolysis temperature, drying plastics at 100 °C.

The oil produced as such from the pyrolysis process cannot be used as fuel, because of its high viscosity and the presence of halogens. The oil must then be refined, after the pyrolysis process. Dehalogenation, for example, allows the elimination of halogen-containing organic compounds: through cracking, with the aid of hydrogen-rich compounds, the halogen-carbon bonds are broken and finally removed from the pyrolysis oil as harmless halogen salts (https://www.umsicht-suro.fraunhofer.de/en/Our_Solution/chemical-recycling.html). As an alternative to refining, it would be necessary to remove the PVC during the collection and sorting phases.

Subsequent studies on pyrolysis oil must focus on reduction of its viscosity and stabilization of its physical and chemical characteristics, such as cetane index, pour point, acid number, etc..., so that they fall within the expected ranges of values from engine requirements, in compliance with the emission limits in force (such as the ISO 8217 standard).

In this way, by exploiting the energy supplied by the pyrolysis products for the operation of the ship's engine, the pyrolyzer, the flue pipes and the heat exchanger, the ship would be energetically self-sufficient.

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