Thermochemical Processes

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A few polymer types preferentially decompose to their monomers in chemical processes. This so called depolymerization is seen for polycondensates, as well as for PMMA, PS, and PTFE. Basically, pure polyesters like PET (just like polyamides and polyurethanes) can be depolymerized chemically by solvolysis. Thermal degradation of polystyrene yields styrene monomer and its derivatives when the heating rate and reactor residence time are moderate, and a similar reaction is observed for thermal decomposition of PMMA and PTFE. All other polymers decompose statistically, or initially form non-monomer products that are favored by the molecular structure, which results in a broad product spectrum. Also, process parameters such as temperature have a tremendous impact on the distribution of thermochemical degradation products because of chemical attack of intermediate species and subsequent reactions during radical-based decomposition. Key aspects of the degradation of pure polymers, and their products, are explained in more detail in Chapter 2 and Section 9.1.1).

When co-polymers, polymer mixtures, or polymers accompanied by other reactive components like biomass or halogens are to be recycled chemically, intermediate species interact during degradation. Due to modified degradation mechanisms, chemical recycling of mixtures results in a product spectrum that is different from that of pure polymers. Furthermore, plastic products themselves contain non-plastic additives, which can also be reactive to a large extent. Examples are fillers like lime, titanium white (and even carbon black), stabilizers, flame retardants, and polymerization catalyst residues. In addition, both process parameters and technology determine recycling product yields and quality.

As real waste rarely consists of pure substances, waste recycling processes striving for the pure polymer or the monomers have to deal with issues resulting from this fact. Waste composition and properties can hamper its processing, and in any case, mechanical recycling of plastics results in lower product quality than virgin polymer. Thermosets, elastomers, and composite materials cannot be recycled exclusively mechanically, but solvent extraction as well as solvolysis of suitable polymers requires well-defined waste feedstock. Thermochemical recycling basically

overcomes these issues by transforming mixed waste into feedstock for new products with virgin quality. To achieve this, thermochemical processes must decompose polymers and non-plastic hydrocarbons to petrochemical feedstock as well as destroy contaminants and remove byproducts.

This chapter outlines thermochemical recycling processes for plastic waste. Key technologies are described and are integrated into full waste-to-chemicals process chains. Section 11.1 briefly covers the basic requirements of processing plastic waste, followed by Section 11.2 and Section 11.3 on the most important thermochemical recycling technologies and well-developed application examples. The aspect of mass and energy balance is emphasized, where this information is available, in order to address process-efficiency and provide data for sustainability evaluation. Section 11.4 gives an outlook on the application of technologies to the thermochemical recycling of mixed plastic waste. Parts of the technology subsections have been published elsewhere [1, 2].

11.1 Challenges and Approaches

11.1.1 Plastic Waste

The challenge of plastic waste recycling results from the nature of waste. It increases with the difficulty in collecting and sorting a specific polymer, and with the grade of functionalization of a specific plastic product. Mechanical recycling can be applied to thermoplastics that can be recovered as pure polymer, but any ingredient of the polymer product impacts the recyclate quality. Dissolution provides opportunities to physically recover polymers from higher functionalized plastic products with a defined (but not too complex) composition, including simple composite materials. The situation is similar for solvolysis, insofar as the polymer type is suitable to be depolymerized by a solvent. Thus, if applicable, solvolysis can recycle even specific thermosets, and as a chemical process it overcomes recyclate quality issues. However, solvent-based recycling processes require a constant waste quality, with each process being limited to a specific sortable product. By this, non-thermochemical plastic waste recycling is successful with post-consumer plastic products specifically from the packaging sector and with well-defined manufacturing residues. Opportunities to expand plastics recycling result from future product design-for-recycling, and from application of mechanical recycling and solvent-based technologies to well-defined post-consumer products that can be collected separately. Applications are given in the respective chapters of this book.

Most plastic waste is not easy to recycle. Many long-life products from the non-packaging sectors are highly functionalized, and post-consumer products appear as

mixed waste with a broad and often unknown specification. In principle, thermochemical recycling can be applied to any such plastic waste, because composites can be thermally destroyed, organic and mineral/metal parts can be detached, and polymers and their ingredients can be decomposed to the level of small molecules that can be separated from each other. Major mixed-plastics waste streams are able to sort residues from lightweight packaging waste (LWP-SR), building demolition plastic wastes such as external thermal insulation composite systems (ETICS), commercial waste (e.g. from the construction sector (CA)), waste electronic and electrical equipment (WEEE), and shredder residues (e.g. from end-of-life vehicles (ASR)). In developed countries, by far the largest plastics waste fraction ends up in residual household waste (RHW). The major component of RHW is of biogenic origin. The plastics fraction in RHW is approximately 10–20%, depending on to what extent separate collection of packaging waste is in place. Table 11.1 gives an overview of the basic characteristics of representative random samples of these heterogeneous wastes.

Table 11.1 Examples of Post-Consumer Mixed Plastic Waste Types and Their Key Polymers

Waste	Main polymers and appearance	Contaminants	Lower heating value [MJ/kg] [2]
LWP-SR	PE, PP, PET, PVC, PS Fluff	See Figure 9.1	25
ETICS	Either EPS, or XPS, or PUR Composite foam	Mortar, plastering, polymer fiber, flame retardants	34
	PE, PP, PVC Bulky, heterogeneous	Mineral and metal adherence, paper, cardboard	34
WEEE	Styrene copolymers, PVC, PP, PE, PA, PUR Mixed fine fraction	Fillers, color pigments, metal fragments, flame retardants	23
ASR	PP, PE, PA, PUR, PVC Mixed fine fraction	Fillers, color pigments, fiber, flame retardants	31
RHW	(all) Bulky, heterogeneous	Organics, sand, metals, glass, etc.	10

* CW = commercial waste.

Any of these wastes consist of more than just hydrocarbons, which are the desired products of petrochemical feedstock recycling. This causes the "heteroatom challenge" - meaning that species containing nitrogen, oxygen, sulfur, chlorine, bromine, and other heteroatoms have to be removed from the recycled products during processing.

11.1.2 Process Parameters

Product yields from chemical recycling are impacted by the feedstock, namely the amount of reactive substances present, its morphology and phase of matter, the process parameters, and the uniformity of reaction conditions, as decided by the respective technology. Briefly, the important impacts of process parameters are:

- **Stoichiometry:** Whilst pyrolysis is a thermal degradation process with indirect heating of the reactants without additional oxidizers, gasification processes are directly heated through partial combustion of the feedstocks by the oxidizer added to the reactor. The oxidizer content in the system must be sufficient to convert all carbon to its monoxide.
- **Minimum temperatures** are required during pyrolysis to allow for full thermal degradation of the specific polymers as well as other reactive components, typically between 350 and 450 °C. Thermodynamic equilibrium of gasification typically requires temperatures above 750 °C to assure full degradation of hydrocarbon species.
- **Minimum dwell times** in pyrolysis and gasification are determined by the reaction kinetics of degradation and partial oxidization, respectively. Reaction times strongly depend on temperature. At higher pyrolysis residence times, secondary reactions occur, which (i) cause further degradation of large molecules and (ii) promote formation of aromatics as well as char.

As mentioned above, the presence of catalytically active species or radical-forming reactive species, as well as added catalysts, can speed up reactions, such that smaller molecules are formed earlier and product distributions change. A similar situation can arise to form aromatics and char during pyrolysis.

Both pyrolysis and gasification are typically controlled by heat and mass transfer. Thus, mixing and back-mixing in the reaction system have a large impact on the reaction conditions and their distribution and uniformity. Typically, due to restrictions in heat transfer, residence times in pyrolysis reactors are longer than indicated by the end pyrolysis temperature. Gasifiers, on the other hand, require higher than kinetically indicated temperatures to overcome the limits of mass transfer. These non-idealities in most cases result in broad product distributions, and are strongly dependent on the specific technology.

11.1.3 Flexible Thermochemical Technologies and Technology Readiness

To be technically and economically viable, these thermochemical conversion technologies have to be integrated into waste processing chains. Thus, the full process chain (see Figure 11.1) usually consists of the (mechanical) pretreatment, the

(thermochemical) conversion, and the cleaning (upgrading) of the main product. Mechanical and chemical recycling get combined and interlocked such that the waste-to-chemicals process chain gets optimized.

Figure 11.1 Process chain for the thermochemical recycling of wastes to secondary (chemical) feedstocks

Because of its chemical composition, mixed waste causes operational challenges for any thermal conversion system – for example, different types of corrosion. In addition, the physical nature of waste complicates mechanical feeding into such systems. In order for mixed plastic waste to be used in such systems, it usually needs pretreatment to remove non-combustible materials, homogenization to minimize operational variations, and ideally transformation so that its physical nature is compatible with mechanical feeding systems.

Mechanical pretreatment can allow waste to meet the physical and chemical specifications required of a specific thermochemical conversion technology. The pretreatment processes are relatively straightforward and involve several stages of sorting, separating, size reduction, and in some cases, biological treatment. Capital costs for the pretreatment systems are moderate and generally worth the benefit of making a low-cost, readily available feedstock stream available. The overall economic analysis is therefore favorable, but viability depends strongly on received gate/tipping fees associated with collecting the municipal waste.

Specific thermal treatment technologies have different feedstock quality requirements for the pretreatment step (mainly particle size, heating value, and chemical composition). This will be referred to in the sections with the specific technologies.

During the thermochemical degradation process, the heating rate, process temperature, stoichiometry, and residence time parameters must be optimized to maximize product yields and process reliability. Often, upgrading of the conversion products is necessary to meet the secondary feedstock specifications. Process chain byproducts can be valuable substances (e.g. metals) or energy carriers (e.g. heat), as well as material streams to be disposed of (e.g. ashes, wastewater).

As described in the literature, process suppliers or operators use a broad variety of methods and technologies for the feedstock recycling of waste at high process temperatures, which all fall under the categories of pyrolysis or gasification. An outline of today's high- and low-technology readiness level (TRL) chemical recycling technologies is given in Section 9.1.1.

So far, most technologies have not had their applicability proven on the industrial scale over a longer period, and so their TRLs are relatively low. Hereafter, we consider those thermochemical recycling technologies that have (a) reached a high TRL *and* size, and that have therefore been able to operate in a steady state over a longer period, by referring to industrial applications (TRL 7 – demonstration system, TRL 8 – first-of-a-kind commercial system, TRL 9 – full commercial application), or (b) only reached a lower TRL (TRL 4 – lab-scale validation, TRL 5 – large-scale prototype, TRL 6 – prototype system), as for current efforts to bring a residual part of the recycling process chain to an industrial scale. This is specifically the case for the important upgrading steps necessary to guarantee the qualities of the products for further use in the process industry.

11.2 Gasification for Chemical Recycling as Petrochemical Feedstock

Gasification in the context of waste recycling is done with the intention of converting biogenic energy resources and anthropogenic waste fractions (e.g. residual biomass, refuse-derived fuels, plastic wastes) into a high quality syngas containing the chemical building blocks H_2 and CO. Syngas is an industrial feedstock used globally for the synthesis of chemical intermediates and products for direct sale on the market. Use of syngas energy by combustion, or as feedstock of synthetic fuels is not regarded as "recycling" per se. Biogas, a mixture of CO_2 and CH_4 originating from biological treatment of organic waste, is not in the scope of this chapter.

Historically, gasification emerged as a sub-process of pyrometallurgy, whereby oxidized metals and coke typically react to form the desired raw metals and lean gases, which are used energetically. Dedicated gasification technologies became industrially relevant in the second quarter of the 20th century in order to avoid overdependence on petroleum imports. Both mobile wood-gas carburetors and industrial-scale coke-based syngas generators were developed up to high TRL. Although not being competitive on the world market, coal-based synthetic fuels were of specific relevance for closed economies such as in South Africa, and significant capacities were installed and operated over a long period (and are still in use). These developments form the basis for feedstock recycling of waste. The technological challenge of reliably producing clean syngas for fuel synthesis is similar to that of chemically recycling waste via gasification.

A second development that assisted gasification technologies came from the oil crisis in the 1970s. Then, large-scale, high-quality syngas production and high-temperature gas cleaning were developed to feed gas turbines in ultra-efficient power

plants of the "integrated-gasification, combined-cycle" type. For reason of energy market prices, most of these large-scale demonstration plants have been closed. Since then, application of gasification to generate second-generation synthetic biofuels has been brought to the demonstration scale. Here, highly developed gasification technology is applied to heterogeneous, pretreated feedstock that is closer in nature to conventional waste. Furthermore, attempts to generate more bioenergy-based electricity has resulted in the installation of several hundred small-scale and easy-to-run decentralized gasifiers, mainly in Europe, during the past decade, but these have lower overall process efficiency. Hydrothermal treatment of biomass is not of significant relevance, so far.

Waste gasification on an industrial scale was developed for energy utilization as an alternative to state-of-the-art incineration technology. For more information, a detailed review of waste gasification is available [3].

11.2.1 Process and Technologies

Any combustible solid feedstock undergoes the consecutive physical and chemical conversion steps of drying, evaporation, char gasification and gas phase oxidization of combustibles during a thermal process with presence of an oxidant. Depending on the zonal segmentation of the reactor interior, the intermediate product flows between the zones, and the conversion steps occurring, final syngas quality can vary significantly. In the overall gasification process, sub-stoichiometric addition of oxidant will (theoretically) be sufficient to convert all char. In high-temperature gasification, relatively-clean syngas can be generated, and thermodynamic equilibrium can be achieved by introducing additional oxidant; the price of this is the part of the feedstock needed for combustion to produce CO_2 and H_2O. If lower process temperatures are applied, the synthesis gas yield is higher, but it contains problematic non-equilibrium components such as trace-gas species, residual methane, and high-boiling hydrocarbons (tars).

There are many design variants for industrial-scale gasification. For the gasification of carbonaceous solid fuels, fixed-bed, fluidized-bed, and entrained-flow gasification processes have been developed; the latter is also particularly suitable for (viscous) liquid feedstocks. As a rule, fixed-bed gasifiers require coal as an additional feedstock when used on biomass or waste, as they have bed structural requirements similar to those of a blast furnace. With regard to the achievable synthesis gas quality (residual methane and tar contents), entrained-flow gasifiers are outstanding, fluidized-bed gasifiers (operated in either circulating or bubbling mode) follow thereafter, and fixed-bed gasifiers (especially in counter-current operation) deliver the lowest qualities. Conversely, the pretreatment cost for feedstocks of an entrained-flow gasifier are the highest, due to the fact that entrained-flow

gasification requires either liquid or pulverized solid feedstock at a maximum grain size of approximately 0.1 mm. Fluidized-bed reactors can accept up to 50 mm grain size and fixed-bed gasifier's particle size distribution limits are even lower, but a significant part of the feed must show persistent physical and thermal stability to form a stable matrix inside the reactor. The fluidized bed technology is described in more detail in Section 9.1.3.

All three gasifier technologies mentioned above are scalable up to 100 MW in size, but only entrained-flow gasifiers can achieve a 1000 MW size due their high reaction density, specifically when they are pressurized. Entrained-flow gasifiers can operate at up to 80 bar, thus avoiding additional costly syngas compression up to the pressure level necessary for thermal separation technologies, chemical synthesis, or gas turbine fuels, and supporting very high throughput of feed with a relatively small reactor volume. Usually, pressure levels of fluidized-bed and fixed-bed gasifiers are lower.

Today, industrially scalable entrained-flow gasifiers dominate the market for new installations on fossil feedstocks used, and the use of fixed-bed gasifiers is decreasing. Fluidized bed gasifiers have little significance in industrial-scale syngas production, but a large number of small-scale units have been recently applied, mainly for the use of pure biomass in bioenergy.

Long-established metallurgical industry technologies are also used to gasify waste. These include the shaft furnace in the production of non-ferrous metals, and the blast furnace used in raw iron production. A special form is gasification in a limestone moving bed, derived from the lime industry. The particularly high process temperatures lead to the separation of the feed substances (separation of the molten metallic or mineral fraction). The process gas contains the hydrocarbons in partially oxidized form. Plastic waste in this case replaces part of the coke or the blow-coal, and acts as a reducing agent. The gas qualities are comparable to those of fixed-bed gasifiers. The use of waste as a predominant feedstock is carried out only on the medium-scale for vitrification of slags and for the energetic use of the synthesis gas, especially in Japan [4], and has reached a high TRL. The requirements for waste quality and permissible waste components are higher than those of the above-mentioned types of gasifiers, whereas the raw gas qualities are lower due to the pyrolysis gas components contained; resource and energy efficiency are also lower.

Even higher temperatures are reached when the energy is provided electrically, to generate a reactive plasma. Thermodynamically, there are only slight advantages in the gas composition over, for example, entrained-flow gasification. The external energy consumption is particularly high. The application of plasma gasification has so far mainly been carried out on hazardous waste at a small scale.

11.2.2 Syngas Upgrading – Cleaning of the Raw Synthesis Gas

Waste feedstock recycling by gasification requires an exceptionally high syngas quality due to the subsequent catalytic synthetic processes. In fact, the quality needs to be higher than for energetic utilization of syngas in turbines for power generation. Nitrogen dilution as well as hydrocarbon content of the syngas has to be avoided, and so the upstream gasifier has to be operated with oxygen or steam as gasification agent, similar to combined heat and power (CHP) applications. The raw synthesis gas coming from the gasifier must be purified and conditioned to meet the required specifications of, for example, methanol synthesis. In particular, the limit on concentrations of trace gases containing sulfur, halides, and nitrogen are much lower than for combustion exhaust gases, and a substantial proportion of particles must be removed. Small- and large-chain hydrocarbons must also be removed from the syngas. In addition, the H_2/CO ratio must be chemically adjusted depending on the downstream process of interest.

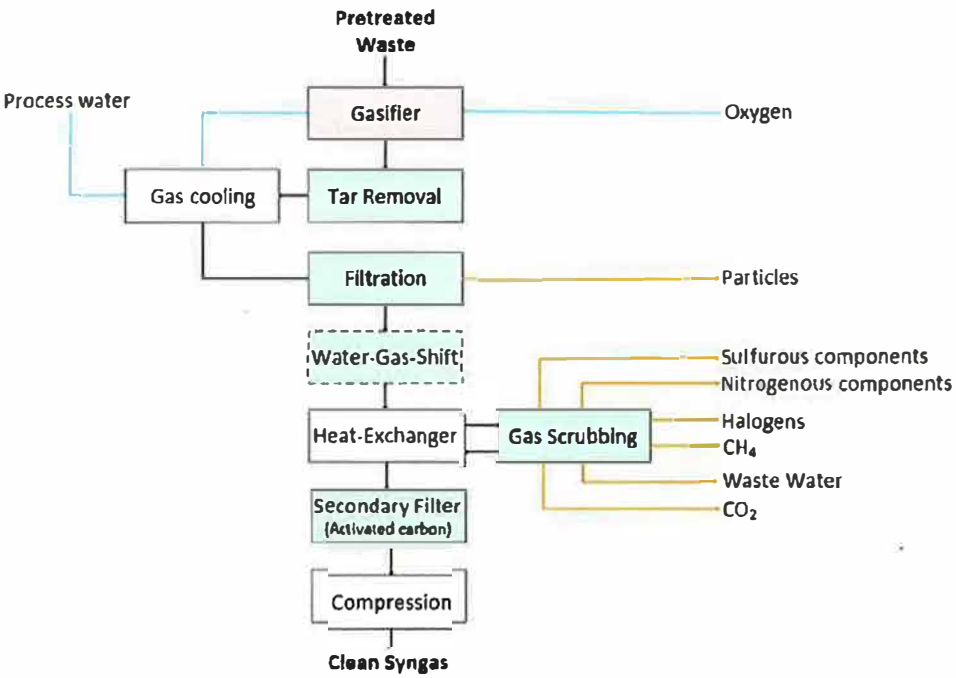

Figure 11.2 Major material flows and process steps in syngas production by gasification of pretreated waste

Figure 11.2 shows the necessary process steps in the synthesis gas treatment chain. It contains heat recovery steps to enable overall autothermal processing and high energy efficiency. Tar removal steps have to be integrated at specific conden-

sation temperatures in order to manage plugging. This is the key technology issue that places limits on the feedstock, as well as limiting the load flexibility of the plant. Following removal of particulates by (for example) high-temperature filtration, a water-gas-shift reactor is included to adjust the H_2 and CO content over a suitable temperature range, followed by a multi-component gas scrubbing technology, placed upstream. Finally, compression is carried out to achieve the required methanol reactor pressure.

It has to be pointed out that this gas treatment and conditioning effort is significant in determining the processing cost of waste feedstock recycling. Furthermore, raw syngas quality critically depends on the gasification technology applied. Thus, the cost of raw gas treatment in the case of entrained-flow gasification would be much lower than in the case of fluidized-bed gasification, which ranks above fixed-bed gasification. On the other hand, the feedstock requirements of the gasifier and thus pretreatment cost vary with technology. Therefore, fluidized-bed gasification, having the lowest requirements, ranks above fixed-bed gasification and entrained-flow gasification. Furthermore, fixed-bed gasification requires a stable coke particle reacting bed, typically achieved by adding fossil coke to the waste feedstock.

11.2.3 Large-Scale, High-TRL Gasification Applications for Chemical Recycling

Gasification as part of a thermochemical recycling process aims at providing secondary feedstock from waste for C_1-chemicals such as methanol and C_1-based value chains such as methanol-to-olefins. For our purposes, Syngas-based fuel synthesis processes such as the Fischer–Tropsch synthesis are not considered as recycling; however, as they contribute significantly to greenhouse gas emissions when fed with natural gas, there is an opportunity for waste to contribute to emissions reduction in that sector, too.

Industrial gasification today mainly uses entrained-flow gasification, with many installations in the beginning of the 21st century for gasification of pulverized coal. All gasification developments of the 20th and the beginning of the 21st were driven by local availability of cheap carbon-rich feedstocks. Usually, these gasifiers are oxygen- and steam-blown, in order to avoid nitrogen dilution in downstream gas-processing plants and to increase process efficiency.

The first applications of gasification technologies for waste feedstock recycling were driven by attempts to recycle plastics in the late 20th century. Specifically in Germany, two industrial-scale gasification technologies on the 100 MW scale were operated over a longer period with different wastes (and mixtures of them) in order to produce methanol. In both cases, existing technologies for lignite feedstock developed to avoid overdependence on oil imports were adapted to process refuse-

derived fuel (RDF) resulting from municipal solid waste (MSW) or even collected packaging plastic waste.

At the German Berrenrath plant, a high-temperature Winkler (HTW) gasifier was run on lignite, RDF and plastics using oxygen, steam, and CO_2 as gasification agents. This gasification technology is characterized by a higher freeboard temperature of approximately 950 °C, located above the bubbling fluidized bed at 10 bar. This results in a better syngas quality. With a heat load of 175 MW, it fed a 100 kt/a methanol plant. The plant was shut down after 10 years of successful operation in the mid-1990s, for economic reasons, mainly methanol prices.

At the Sekundärverwertungszentrum Schwarze Pumpe (SVZ) in Germany, which was already co-processing waste, sewage sludge, and lignite in revamped fixed-bed Lurgi gasifiers, for the first time a British Gas Lurgi slagging gasifier was installed in early 2000. Different types of waste were converted to syngas in the BGL gasifier at 25 bar, and fed a 100 kt/a methanol plant. The minor syngas quality issues of the fixed-bed gasifier were overcome by the extensive complementary plant structure of this production site, where significant tar quantities removed from the syngas served as feedstock for an entrained-flow gasifier. SVZ operations were terminated in 2005, mainly for two economic reasons: (i) the methanol production cost of this rather small dedicated production site were too high compared to world-scale methanol plants based on natural gas and, (ii) for mixed wastes and sorting residues, SVZ had to compete with cheaper waste incineration, because legal regulations at the time allowed low recycling rates for collected plastic waste material.

Both technologies – HTW as well as BGL gasifiers – could not be operated fully on RDF, but required significant co-processing of char-forming fossil feedstock (namely lignite) and waste. In addition, RDF had to be pelletized before being fed to the gasifiers.

This is not the case for the Showa Denko gasification plant at Kawasaki, Japan, which has been operated since 2004 with an increasing amount of pure collected and presorted polyolefinic packaging waste feedstock, which is pelletized on-site before being fed to the gasifier. Here, a two-stage gasification process is used at 10 bar, which consists of a steam/oxygen-blown bubbling fluidized-bed step at temperatures below 800 °C, followed by an steam/oxygen-blown slagging entrained-flow-like step to guarantee excellent syngas quality at ca. 1500 °C. The syngas is then converted in a water-gas-shift reactor, and hydrogen is removed and fed as part of the feedstock to a world-scale ammonia plant. Due to its specific nature (requiring a high process temperature and not making use of the feedstock's carbon content), the cold gas efficiency and economics of this process is significantly lower than for the aforementioned gasifiers.

At Edmonton, Canada, Enerkem has operated (since 2017) a solid recovered fuel (SRF)-to-methanol process, which is using pretreated residual household waste

(RHW) with a higher content of non-plastic waste of biogenic origin. It is based on its own proprietary technology that has been developed over a longer period and in several projects and plants with different sizes. Further projects have been announced in various countries. Again, this process uses bubbling fluidized-bed gasification technology and staged addition of oxidants. The fluidized bed is operated at below 700 °C due to the low SRF ash melting point, followed by a freeboard at higher temperatures to minimize tar formation. After flue gas treatment and recirculating of the tars to the fluidized-bed reactor, a proprietary methanol-to-ethanol process is applied at a smaller scale. Reactor over-pressure and capacity are smaller than for the processes mentioned above.

A more detailed description of these last three waste-to-methanol processes is available [4].

In addition to dedicated gasification processes, pyrometallurgical processes can be used to chemically recycle waste. Specifically, during processing of iron ore to raw iron, plastic waste is used to replace part of the reducing agent. Constraints here are that the waste feedstock ingredients must not disturb the production of raw iron or subsequent steel manufacture. This excludes many plastic waste types. In blast furnace processes, collected and sorted polyolefinic plastic waste is used to replace part of the coke but not the charge of the furnace. This application of waste feedstock is state-of-the-art and has been successfully applied at the Voest Alpine steel mill in Linz, Austria, for a long time. Feedstock utilization of the diluted syngas coming from these melting-type furnaces has not been applied so far due to the bad gas quality resulting in a high gas treatment and cleaning effort. Recent efforts aim at making chemical use of steel mill gases. Also, using lime shaft furnaces as waste gasifiers has been subject to R&D activities. The TRL of the latter processes is still low.

11.2.4 Balancing of a Gasification-Based Waste-to-Chemicals Process Chain

Gasifiers can be modeled as thermodynamic equilibrium reactors based on the specification of the feedstock composition and of process heat losses. By adding oxygen or steam to the waste feedstock, the necessary reactor temperature and syngas composition are reached with the highest possible theoretical cold gas efficiency. The cold gas efficiency describes the possible synthesis gas yield in terms of chemically bound energy of the feedstock, thus taking into account the effort to achieve the gasification temperature by burning part of the feedstock. Theoretical gasification temperatures can be as low as 750 °C, resulting in high cold gas efficiency. Due to real process limitations, higher temperatures have to be chosen, resulting in lower process efficiency. Nevertheless, for real gasification processes,

process-dependent deviations from thermodynamic equilibrium have to be taken into account by adjusting theoretical syngas composition with respect to unreacted, non-equilibrium hydrocarbon species.

Here, we consider gasification of pretreated MSW in a fluidized-bed gasifier to provide syngas for methanol synthesis. This example case corresponds to a similar fully integrated large-scale process that is operated by Enerkem (see above). Because detailed mass and energy balances that allow process evaluation have not been published so far, the results shown here are based on process simulation that has been reported in detail elsewhere [5]. It includes mechanical pretreatment of the MSW collected by applying state-of-the-art SRF production technology, SRF gasification, and upgrading of the raw syngas to fulfill the demands of catalytic methanol synthesis. For an oxygen/steam-blown gasification of pretreated MSW considered here, the equilibrium balance was calculated at 950 °C. To consider the non-equilibrium syngas tar and methane contents, experience from the literature was taken into account.

Figure 11.3 shows the material flow diagram for this case. The overall process acts to separate the substances, and so recovering the metallic value products, separating off mineral products, and recirculating carbon from waste to syngas. The carbon mass fraction of the synthesis gas as desired recycled feedstock is significantly higher than the plastics carbon mass fraction of the waste. In the case of fluidized-bed gasification, about one third of the initial waste mass is converted into synthesis gas. Based on the carbon used, it is about 46%. All other waste carbon is converted to CO_2 during incineration of the fine mineral residue from mechanical treatment, to achieve the necessary gasification process temperature, and in particular to adjust the required hydrogen content of the synthesis gas via the water-gas-shift reaction during syngas upgrading. Primarily due to the lack of hydrogen in the waste feedstock, roughly half of the waste carbon is recovered when making methanol alone. In the energy balance (not shown here), the thermochemical process part enables energy-independent operation of the entire process chain due to the supply of high-quality process heat, which also takes into account the electricity requirement. The overall process energy efficiency of the chemical recycling process chain can be up to 90%, by making use of both gasification cold gas efficiency and high-value syngas sensible heat.

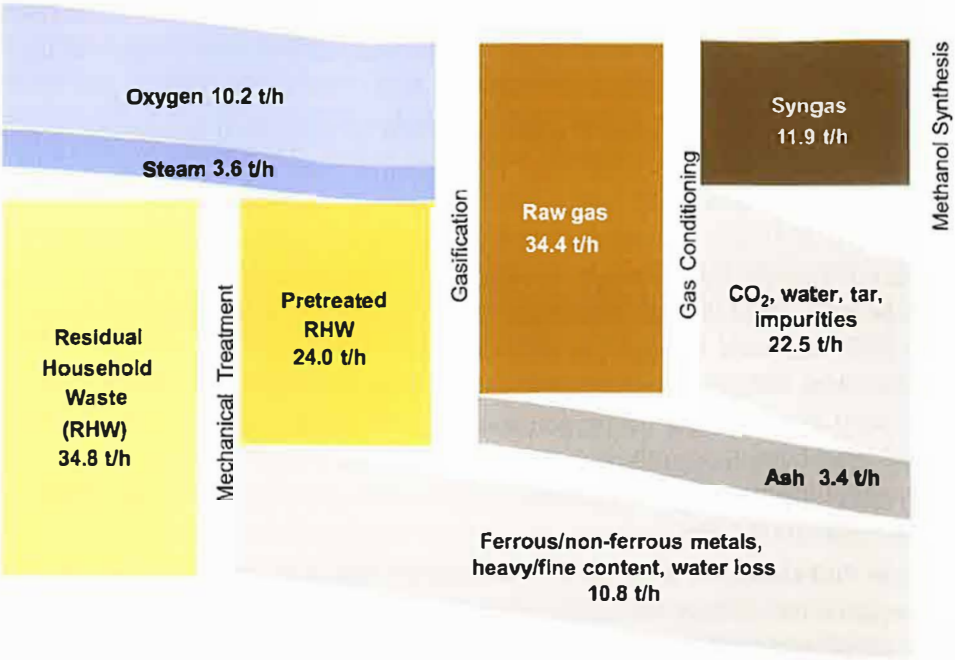

Figure 11.3 Material flows in the process chain for fluidized-bed gasification of residual household waste (RHW) to produce methanol syngas. The heat load of the gasification step is 100 MW

11.3 Pyrolysis for Chemical Recycling as Petrochemical Feedstock

11.3.1 Balancing of a Pyrolysis-Based Waste-to-Chemicals Process Chain

Pyrolysis is a kinetically controlled process. In technical pyrolysis systems, heat and mass transfer control the local reaction conditions within the reactor. Thus, pyrolysis product yields and distributions depend on (i) waste type and composition, (ii) applied pyrolysis and technology, and (iii) the process parameters chosen. Modeling and simulation of these complex multi-phase reacting systems is the scope of research efforts. Pyrolysis process design is thus empirically based and requires experimental data.

The main pyrolysis sub-processes are represented in Figure 11.4. Pretreated waste thermally decomposes in a single- or multi-reactor pyrolysis step, forming permanent gases, condensable vapors, and solid products. A combustion step utilizes the

energy of byproducts and destroys undesired carbon-containing substances, whilst the main product is yielded in a condensation step. Phase separation and cleaning steps included are not shown in this figure, and depend on specific pyrolysis technologies, as do removal and utilization of solid byproducts and residues.

In the case of waste mixtures containing biogenic fractions, as well as oxygen-containing plastics which are considered here, aqueous condensates of low calorific value cannot be avoided. The condensable organic components form the product of value that must fulfill the product specification as secondary chemical feedstock. A key issue is the removal of contaminants such as halogens from the process flow in the reactor. Therefore, either sorbents are added to the reacting mixture in the reactor, or pyrolysis gases and vapors are treated before condensation. Also, extensive post-processing of the liquid pyrolysis products (for example, by hydrogenation) can be necessary to adjust the liquid hydrocarbon composition in the condensable pyrolysis products. Typically, permanent gases and aqueous fluids are burnt to enable the autothermal operation of the overall process. Carbon-containing solid products can be burnt for this purpose, too. Residual mineral fractions have to fulfill landfilling requirements. As the permanent gas might contain hydrocarbons (to a large extent depending on feedstock and process), it might be considered for feedstock utilization, which is not the case in this example.

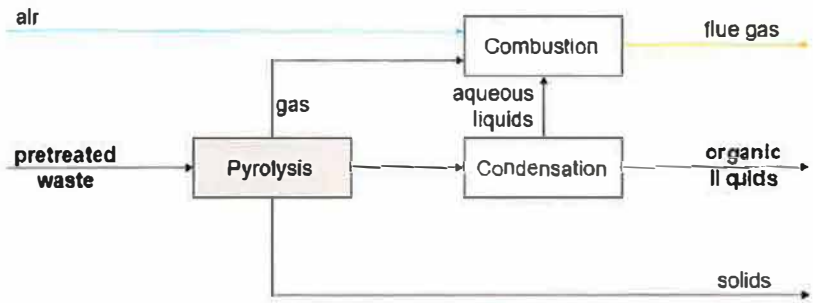

Figure 11.4 Major material flows and process steps in liquefaction of pretreated waste by pyrolysis

Here, we consider non-catalytic pyrolysis of pretreated MSW to provide pyrolysis oil (organic condensate) as a steam cracker feedstock blend. This example case corresponds to the gasification case reported above. Because detailed mass and energy balances that allow process evaluation have not been published so far, the results shown here are based on laboratory-scale experimental data on product yields and distributions for similar waste that have been scaled-up, and have been reported in detail elsewhere [5]. It includes mechanical pretreatment of the MSW collected by applying state-of-the-art SRF production technology, SRF pyrolysis, upgrading of the pyrolysis oil to fulfill the demands of naphtha as steam cracker

feedstock, and energetic utilization of the pyrolysis byproducts. Herein, SRF pyrolysis data were taken from [6].

Figure 11.5 shows the material flow diagram for this case. The overall process acts to separate the substances, recovering the metallic value products, separating off mineral products, and recirculating carbon from waste to organic liquids (pyrolysis oil). The carbon mass fraction of these organic liquids as the desired recycled feedstock is significantly higher than the carbon mass fraction of the plastic waste. In the case of pyrolysis, only about one-fifth of the initial waste mass is converted into pyrolysis oil. Based on the carbon used, it is about 45%. All other waste carbon is converted to CO_2 during incineration of the fine mineral residue from mechanical treatment, to achieve the necessary pyrolysis process temperature, and in particular to burn the pyrolysis byproducts. The H/C ratio of the pyrolysis oil is close to that of heavy steam cracker feedstocks, such that consecutive hydrogenation of the pyrolysis oil is applied to remove heteroatoms and to saturate remaining double bonds rather than to provide a higher hydrogen content. In the energy balance (not shown here), the thermochemical process part enables energy-independent operation of the entire process chain due to the supply of high-quality process heat, which also takes into account the electricity requirement. The overall process energy efficiency of the chemical recycling process chain can be up to 90% by making use of both chemical energy of the pyrolysis oil and sensible heat of the combustible byproducts.

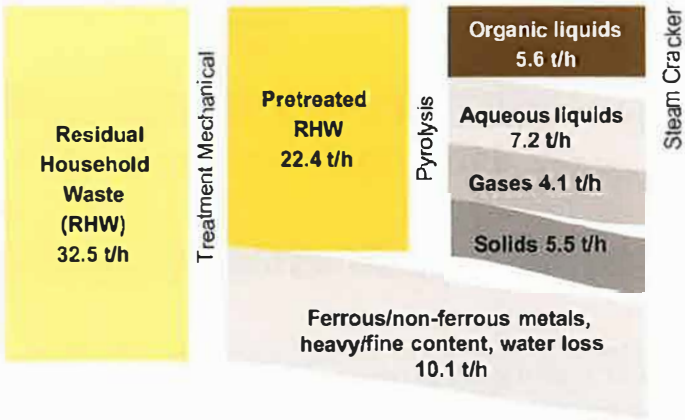

Figure 11.5 Material flows in the process chain for pretreatment and pyrolysis of residual household waste (RHW). The heat load of the pyrolysis step is 100 MW

From this example comparison of chemical recycling of mixed waste by gasification and by pyrolysis, one can see that basically the alternatives perform similarly in terms of indicators related to mass and energy balance. Nevertheless, for pyrolysis this is a theoretical case because no attempts have been made so far to pyro-

lyze MSW-based SRF with a relatively low plastic content at the relevant scale. Waste streams based on plastic with low biogenic content are the focus of this chapter, as they are the waste streams to be recycled chemically in addition to physical recycling processes.

Little comprehensive data can be found in the literature regarding the reproduction of pyrolysis performance for different types of mixed plastic waste, specifically on a technical scale. With respect to the waste situation and composition in Germany about 25 years ago, most widespread investigations were carried out by Kaminsky's group at Hamburg, applying high-temperature fast pyrolysis in bubbling fluidized-bed reactors on a bench- and pilot-scale to different kinds of waste plastics and mixed waste. A good outline of the chemical recycling performance is available [7].

Zeller et al. [2] recently reported the mass and energy balances of pilot scale pyrolysis of important mixed plastic waste streams shown in Table 11.1. Due to the technology and the moderate process parameters applied, in this case plastics decompose quite slowly and all products were collected separately. Processing occurred non-catalytically in a screw-type reactor without further additives, and in an inert atmosphere. Figure 11.6 shows the product mass yields of these investigations.

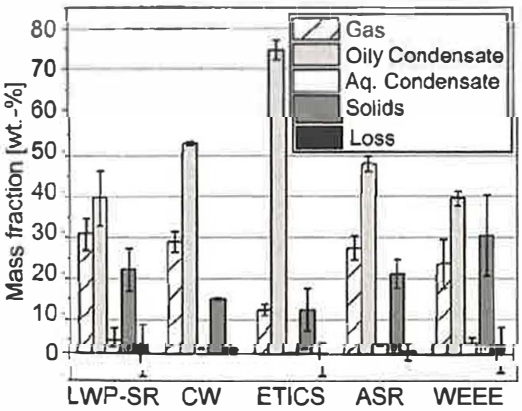

Figure 11.6
Average mass balances for pilot-scale screw-reactor pyrolysis experiments with different mixed plastic wastes at 450 °C and 30 min solids dwell time [2]

The yields of the individual pyrolysis product fractions vary depending on the characteristic composition of the feedstock. Within a specific feedstock, it varies due to inhomogeneities impacting the feed as well as measurement uncertainties. The yield of pyrolysis solids is between 10% and 30%. This product fraction consists of coke and inert materials originating from functionalization of the plastics and contamination of the waste. Thus, the ash content of the feedstock must be considered when interpreting the yield of solids. Between 40% and 75% of the feedstock mass pyrolyzed is found as oily condensate. The thermal insulation system

feedstock shows the highest condensate yield, while the oily condensate yield from WEEE and lightweight packaging sorting residue is the lowest. An aqueous phase with up to 5% of the feedstock occurs as a second condensate phase. Since pyrolysis is carried out with dried feedstock, the introduction of chemically unbound water into the reactor as a source for the aqueous pyrolysis fraction is low. The aqueous condensate is thus attributed to oxygen-containing plastics or biomass contamination. The pyrolysis experiments with ASR and WEEE with an expected content of engineering plastics, as well as LWP-SR with biomass contamination, show an increased release of aqueous product and thus confirm this assumption. The permanent gas fraction amounts to 13% to 30% of the total mass.

The carbon yield refers to the amount of carbon that can be recycled in relation to the waste feedstock carbon fraction. For this purpose, the carbon content of the oily condensates was determined and combined with the oil yield. The pyrolysis oil carbon yields are 51.1% for LWP-SR, 60.0% for CW, 74.6% for ETICS, 57.5% for ASR, and 60.5% for WEEE. Depending on the location and detailed design of a large-scale pyrolysis plant, in addition to the carbon in the condensate phase, the carbon contained in the gas phase could also be partially recycled. Hence, the reported carbon yields are to be understood as minimum recycling rates.

At least 50% of the introduced carbon is recovered in the oily phase and is thus in principle available for reintroduction into a value chain. For ETICS, which contains almost solely polystyrene a (a polymer that thermally depolymerizes), about 75% of the feedstock carbon is contained in the oily condensate. The potential for value chain reintegration of the pyrolysis products must be assessed individually for each waste type based on the specific composition.

Since the waste feedstocks investigated are a mixture of plastics and other materials, detailed statements on the influence of the polymers contained in them, in terms of energy demand, cannot be made. Thus, the determined energy balances are of technical nature and relevant only in the context of a given application. The energy demands for heating, melting, pyrolysis, and evaporation determined in the pyrolysis experiments were normalized using the lower feedstock calorific value. They are 5.1% for LWP-SR, 5.2% for CW, 4.9% for ETICS, 5.4% for ASR, and 3.7% for WEEE, and are thus within a narrow range. In a technical process, heat losses and the energy demand of supporting infrastructure would add to these values.

11.3.2 Process and Technologies

Pyrolysis as a recycling process aims to convert solid carbon from (for example) biogenic residues or plastic waste streams to supply the process industry with easily accessible hydrocarbon feedstocks. Pyrolysis was the first dedicated thermochemical process besides combustion, and evolved primarily for the production of

coke from wood or coal. Left over from this time before the era of crude oil are two industrial-scale applications:

- The coking plant providing coke from hard coal in large fixed-bed furnaces operated batchwise supplies the pyrometallurgical industries. The solid yield is high due to the feedstock. Byproducts (coal tar and coke oven gas) are mainly used energetically.
- The production of charcoal from wood with a yield typically not higher than 50 wt%. Usually, hard wood is pyrolyzed in batch processes in fixed-bed reactors. As byproduct, vapors are condensed and thermally separated to yield (for example) acetic acid and tar oils. Quite a few such applications worldwide supply the charcoal market.

In addition, there are some industrial-scale petrochemical applications operated continuously, such as those designed for pyrolysis of oil sludge in a rotary kiln. Most large-scale coking processes in refineries are operated with oxygen-carrying gases such as steam, thus not strictly falling under the pyrolysis category defined above.

In recent times, pyrolysis was developed further to have a range of applications to various feedstocks and purposes. Examples are the liquefaction of lignocellulosic biomass for the production of substitute fuels and the pyrolysis of residual household waste for energy utilization. Rotary kilns, auger-type reactors and fluidized-bed reactors are applied in order to continuously operate the processes.

Fast pyrolysis has been investigated extensively in recent decades for the purpose of direct thermochemical liquefaction of sustainable biomass. Resulting bio-crudes and bio-oils are upgraded to bio-fuels or bio-chemical intermediates, for example by means of hydrogenation. So far, only a small number of commercial applications have appeared, mainly in the non-transportation fuel sector. IEA Bioenergy [8] has provided an overview of globally installed thermal liquefaction capacities on the demonstration scale [8].

Recently, pyrolysis has came back into focus as an alternative to polyolefinic waste incineration. In addition to the thermochemical conversion step itself, upgrading or product cleaning steps are necessary to guarantee the qualities of the products for feedstock utilization in process industry. Individual pyrolysis technologies use different reactor types. The most important reactor types applied to continuous plastic waste pyrolysis are:

- **Extruders:** As a standard technology in the polyolefin compounding industry, as well as for processing of polyolefin recyclates, twin-screw extruders are applied to degradative melting and pre-conditioning of mixed polyolefinic waste. Herein, the extruder functions as a first reaction step. Limits are: (i) temperature, which commonly is below the total degradation temperature of most polymers, and (ii) heterogeneity, particle size, and content of non-melting components of the waste feedstock.

- **Stirred-tank reactors/flow reactors:** A stirred tank can be used in continuous operation to pyrolyze meltable plastics. It provides the closest to fully homogeneous reaction conditions of all reactor types. Operation limits can be temperature and size, as well as non-melting ingredients. An auxiliary system is needed to provide homogeneous and molten feedstock and to control flow. This system itself can be used as a flow reactor when it processes a large circular liquid flow to which feedstock is added and from which product is separated.
- **Screw reactors:** In an auger-type reactor that is filled to a small extent only, heterogeneous solid feedstock is conveyed in a well-defined manner during conversion, making this technology very flexible in terms of feedstock composition, morphology, and pyrolysis process parameters. Throughput is limited due to mechanical design constraints, so that scale-up to the t/h of waste scale requires multiple machines.
- **Rotary kilns:** A rotary kiln is the most flexible machine to process any morphology of solid feedstock for thermal decomposition. Furthermore, for purpose of pyrolysis there are no temperature limits and scale-up can lead to high throughput of waste material. Whilst solids mixing and residence time control are well-assured, vapors and gas residence time in the large freeboard are less well-defined. Operational issues can result from sticky materials and from ensuring that the drum sealing of the large rotating machine remains leakproof under pyrolysis conditions.
- **Fluidized-bed reactors:** A bubbling fluidized-bed reactor applied to mixed plastic waste pyrolysis functions as a continuous stirred tank reactor but can be used for non-melting material, too. Furthermore, for the purpose of pyrolysis there are no temperature limits, and scale-up can lead to high throughput of waste material. Operational issues result from the higher demands in terms of feedstock granularity and its homogeneity. Furthermore, the stickiness of meltable polymer waste has to be controlled well to assure operability of the fluidized bed. Whilst reaction conditions can adjusted very well to optimize product yield and composition, the need to fluidize the material typically requires recirculation of post-processed pyrolysis gas, affecting the reaction conditions in the reactor. So far, there have been no circulating fluidized-bed applications for large-scale plastic waste pyrolysis.

Due to the nature of waste, most technologies require pyrolysis product upgrading. In-situ upgrading includes addition and removal of sorbent species to remove heteroatoms, addition and removal of catalysts in polyolefin pyrolysis to adjust chain length of hydrocarbon species (and thus liquid and gaseous product yields and compositions), removal of particulates from condensable species, and control of oily and aqueous condensation to avoid re-polymerization. Ex-situ upgrading in particular includes catalytic hydrogenation of pyrolysis oil at higher pressure to saturate C-C bonds and to allow removal of heteroatoms from the oil. In addition,

thermal separation technologies might be applied to recover high-value species from the oil and to adjust the boiling curve. Permanent pyrolysis gases might be treated by adsorption or thermal separation of specific heteroatomic species. Finally, solid pyrolysis residues might be fractionated mechanically to recover high-value products or to segregate streams for disposal.

11.3.3 Large-Scale High-TRL Pyrolysis Applications for Chemical Recycling

Pyrolysis of mixed plastic waste has reached the same high degree of maturity that thermochemical technology already has, with the exception of pyrolysis product upgrading and feedstock utilization.

Mixed plastic waste pyrolysis was operated successfully on an industrial scale for a significant period at the MSW pyrolysis plant at Burgau in Germany (1983–2016). MSW and sewage sludge were pyrolyzed in two rotary kiln lines of ca. 2 t/h capacity each, followed by combustion of the pyrolysis gases and vapors in a steam boiler. According to Quicker et al. [9], the electrical efficiency of the plant did not exceed 22% due to the high amount of char in the solid residues. The plant was finally shut down because of unfavorable economics, due to the low electricity revenues as well as the high disposal cost of the solid residues.

Further successful operational cases for this kind of technology, which use the pyrolysis products energetically in combination with combustion and ash melting, are documented for Japan [4]. In Japan, separation and melting of the slag was the focus of MSW treatment for a long period due to legal requirements. This encouraged application of thermal treatment technologies alternative to incineration on the grate, although the energy efficiency and economic performance of the alternatives was worse.

A mature example of chemical recycling of waste by pyrolysis is the end-of-life tire pyrolysis facility at Hirohata steelworks in Japan. Operating since 2004 with a capacity of ca. 4 t/h in each of the two rotary kilns, shredded waste tires are processed to different valuable products: pyrolysis gas, light and heavy pyrolysis oil, carbon black, metals, and a mineral residue. Whilst the latter is deposited and the pyrolysis gas is energetically used to balance the heat demand of the pyrolysis plant, the other products are used in the steel mill as reducing agents and as carbon feed for metal recycling. Chemical-grade quality of pyrolysis gases and condensable vapors is not the focus of this process. Today, waste-tire-pyrolysis R&D and project activities focus on recovery of the carbon black pigment from the solid pyrolysis residue, in sufficiently high quality that it can be re-reprocessed as recycled carbon black in synthetic rubber.

Most recent activities on chemical recycling of waste by pyrolysis all focus on feedstock utilization of polyolefin plastic waste. Driven by the first EU plastics strategy [10], which sets mandatory recycling quotas for the EU countries, plastics producing industry and waste management companies announced several ambitious projects which will lead to capital investment into plastics liquefaction (see Section 9.1.1). The main purpose is to recover end-of-life plastics as petrochemical liquid feedstock. So far, only two plants (in Almeria and Seville, Spain, operated continuously by Plastic Energy Ltd) fulfill this task at a commercial scale. Both operate on pre-selected polyolefinic feedstocks that, amongst others, are almost free of chlorine, biomass, etc.

As laid out above, the waste streams available at industrially-relevant quantities for chemical recycling are not the pure, sortable plastics that are often amenable to pyrolytic processes but sorting residues that are mixtures of anthropogenic products and biomass. Pyrolysis of these waste streams is more troublesome, and the subject of research and development. Alternatives to rotary-kiln technology are considered in order to homogenize pyrolysis reaction conditions and to achieve high-value product yields adapted to the respective waste feedstock. Current R&D efforts aim at increasing the technology readiness level of pyrolysis processes in order to improve the economics of waste pyrolysis. In the case of mixed plastic waste, the technological challenges are the adjustment of product specifications both in the reactor and by post-treatment, and in managing the plugging or sticking tendency of the intermediates within the reactor and in the downstream gas treatment and condensation steps. As references for waste pyrolysis, the few examples mentioned above are not sufficiently well-documented to allow closure of the mass and energy balances and independent evaluation.

11.4 Outlook

Due to the mixed nature of waste and consequent variation in behavior, achieving the desired recycling quota and promoting the circular economy for carbon in the medium-term requires both waste collection and mechanical sorting systems, and chemical recycling of waste. Chemical recycling complements mechanical sorting by overcoming the downcycling issues of mixed materials and by avoiding final incineration of large amounts of separately collected wastes. Combined mechanical and chemical recycling thus maximizes the recycling rate and minimizes the greenhouse gas emissions. For this purpose, the thermochemical processes of pyrolysis and gasification function as thermal separation processes for mixed wastes including plastics, biomass, and mineral fractions.

Whereas sorting technologies are ready to be implemented in order to increase waste recycling, thermal technologies for chemical recycling still need R&D efforts, specifically concerning the flexibility to process varying complex mixed feedstocks and the scale of processing. Alternative thermal waste treatment technologies applied to waste-to-energy are mostly not competitive with waste incineration due to higher process complexity, and the associated need for specific capital investment. In contrast, pyrolysis and gasification processing costs are of the same order of magnitude as waste incineration gate fees when being applied to complex waste feedstock recycling, because of the recycled product revenues (for example, see López et al. [6]). Accordingly, alternative thermal treatment of waste seems economically feasible, and supports the transition to a circular economy.

The balancing of pyrolysis- and gasification-based entire waste-to-chemicals process chains shows that both thermochemical processes perform similarly. Gasification recycles waste carbon to feedstock for C_1-chemistry. It can be applied centrally on the chemical production site or the petrol refinery site, and it requires high capital investment. Pyrolysis, more directly than gasification, recycles plastics to plastics because it converts a significant part of waste to feedstock for monomer production. This is no value by itself. The pyrolysis plant can be decentralized when the focus is on the organic liquid product only. Pyrolysis requires lower capital investment than gasification. This might explain current the chemical recycling focus on pyrolysis technologies.

Comprehensive life cycle analysis can indicate whether use of a specific recycling technology is advantageous for a specific waste. The highest efficiency of chemical recycling in terms of greenhouse gas emissions is achieved when a high amount of chemical energy is recycled and external energy demand is minimized. Guiding principles for process selection can be:

- Choose a recycled product specification that best fits your waste specification:
  - Maintain the H/C ratio
  - Consider the degradation mechanism of the polymers in the waste
  - Stay flexible with regard to waste specification and its variation
- Choose a recycling technology that maximizes the value of the recycled product:
  - Apply mechanical recycling and solvent-based processes to purely sortable and little-contaminated products
    Apply thermochemical recycling to hard-to-sort mixtures, to composites, and to polymers that are easy to upcycle thermochemically
  - Consider central large-scale thermochemical conversion to use gaseous products
- Optimize the entire value chain:
    Adapt pretreatment process to the demands of chemical conversion technology

- Remove as many contaminants and heteroatoms as possible in-situ during conversion
- Try to integrate product upgrading into central structures

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