

Method for Extracting a Wax
from a Pyrolysis Residue

The invention relates to a method for obtaining a wax from a pyrolysis residue.

It is known from the prior art to separate a wax from a starting product by crystallization. For example, US 2002/0096451 A1 describes a method for purifying a petroleum product. A solvent is added to the petroleum product at room temperature. The obtained mixture is then cooled to crystallize and separate a wax contained in the petroleum product.

A similar method for dewaxing is disclosed in WO 2021/115982 A1, where a solvent is added at room temperature to a pyrolysis residue obtained from the pyrolysis of a plastic. The obtained mixture is then cooled to separate a wax contained therein by crystallization.

Further methods related to the separation of waxes are known from US 3,720,599 A, US 5,006,222 A, US 2,614,065 A, WO 2017/168165 A1 and WO 2021/115982 A1.

In the methods known in the prior art, the separation of the wax serves predominantly for the purpose of purifying the starting product. The separated wax may have a relatively low purity. However, for numerous applications, it may be necessary to use a wax with a high purity, which may require further, elaborate cleaning of the separated wax. There is a need for inexpensive, simple methods for the production of a wax with a high purity. It is an object of the invention to provide such a method.

The method according to the invention for obtaining a wax from a pyrolysis residue comprises the steps

(a) providing the pyrolysis residue, wherein the pyrolysis residue comprises the wax,

(b) mixing the pyrolysis residue with a solvent at at least 30 °C to obtain a mixture, wherein the wax is at least partially dissolved in the solvent,

(c) cooling the mixture to crystallize at least a portion of the dissolved wax,

(d) separating at least a portion of the crystallized wax from the mixture.

The invention is based on the finding that a pyrolysis residue, in particular a pyrolysis residue obtained by plastic pyrolysis, is a valuable source of a wax. By mixing the pyrolysis residue with the solvent at at least 30 °C, the wax can dissolve to a large extent or even completely in the solvent and subsequently be separated from the pyrolysis residue with a high purity. The wax obtained can be a sustainable alternative to wax obtained directly from petroleum.

The pyrolysis residue can be obtained by pyrolyzing a plastic, in particular a waste plastic. The pyrolysis can take place in a pyrolysis reactor, preferably at a temperature of 300 to 500 °C, in particular 350 to 450 °C. As a result, a good balance between cost-effectiveness and method efficiency can be achieved.

The pyrolysis may be thermal pyrolysis (i.e., thermal cracking, without the addition of a catalyst) and/or catalytic pyrolysis (i.e., catalytic cracking). Thermal pyrolysis is preferred to avoid any contamination of the wax and/or solid due to catalyst components.

The pyrolysis can be carried out largely in the absence of oxygen, in particular in an inert atmosphere, for example under nitrogen. Due to a lack of oxygen or exclusion of oxygen, complete combustion can be prevented and a polymer contained in the plastic can be cleaved or depolymerized.

Within the scope of the invention, it has proven advantageous if a fraction, preferably a heavy fraction, of the pyrolyzed plastic is used as the pyrolysis residue. In a preferred embodiment, the pyrolysis residue is therefore obtained by pyrolyzing a plastic and separating at least one fraction, preferably a gaseous fraction, from the pyrolyzed plastic. The separated fraction preferably has a lower boiling temperature (or a lower boiling range) than the pyrolysis residue.

In a particularly preferred embodiment, the pyrolysis residue has a boiling temperature (or a lower end of a boiling range) of at least 100 °C, preferably at least 150 °C, more preferably at least 200 °C, more preferably at least 240 °C,

more preferably at least 270 °C, more preferably at least 300 °C. The pyrolysis residue preferably has a boiling temperature (or a lower end of a boiling range) in the range from 100 °C to 700 °C, preferably from 150 °C to 600 °C, more preferably from 200 °C to 500 °C, more preferably from 240 °C to 460 °C, more preferably from 270 °C to 430 °C, more preferably from 300 °C to 400 °C. It has been found that fractions with such a boiling temperature can have particularly high proportions of wax and are therefore particularly suitable for the method according to the invention. It has proven to be particularly advantageous if the pyrolysis residue is a spindle oil, preferably with a boiling temperature (or a lower end of a boiling range) in the range from 300 °C to 400 °C. In this case, the wax content of the pyrolysis residue may be, for example, about 50 % by weight.

The boiling temperature (or boiling range) can preferably be determined by means of the standard ASTM D7500-15:2019. Alternatively, the standard ASTM D2887-22:2022 may be used.

The plastic preferably comprises a polyolefin and/or a polystyrene (PS), wherein the polyolefin may comprise a polyethylene (PE) and/or a polypropylene (PP). The plastic preferably comprises the polyolefin and/or the polystyrene in an amount of at least 65 % by weight, more preferably at least 70 % by weight, in particular at least 90 % by weight, based on the total weight of the plastic. As a result, a pyrolysis residue can be obtained which comprises a significant proportion of an aliphatic hydrocarbon (or a mixture of a plurality of aliphatic hydrocarbons), i.e. a significant proportion of the wax.

The plastic preferably comprises at least 20 % by weight of the polyolefin, more preferably at least 50 % by weight, even more preferably at least 70 % by weight, in particular at least 90 % by weight, based on the total weight of the plastic. As the proportion of polyolefin increases, the amount of wax obtained by the method according to the invention can be increased, which can improve the cost-effectiveness of the method.

In the course of the invention, it has been found that a high polyethylene (PE) content is particularly advantageous, since a particularly high wax content can be obtained during the pyrolysis of plastic with a high PE content. In a particularly

preferred embodiment, the plastic therefore has a PE content of at least 10 % by weight, preferably at least 20 % by weight, more preferably at least 30 % by weight, more preferably at least 40 % by weight, more preferably at least 50 % by weight, more preferably at least 60 % by weight, even more preferably at least 70 % by weight.

The plastic may comprise a further polymer from the group consisting of thermoplastics, duromers and/or elastomers, in particular an acrylonitrile-butadiene-styrene copolymer (ABS), a polyvinyl chloride (PVC), a polyamide (PA) and/or a polyester.

Before pyrolysis, the plastic can be plasticized, for example in a mixer, in particular in an extruder. The plastic is preferably heated to a temperature of at least 120 °C in order to plasticize it, more preferably to a temperature of 200 to 500 °C, even more preferably of 400 to 470 °C. Then the subsequent pyrolysis can be carried out more energy-efficiently and in a shorter time. In the extruder, the plastic can also be degassed to produce a uniform mass without gas inclusions, so that a homogeneous pyrolysis product can be obtained by the subsequent pyrolysis.

Before the pyrolysis, a diluent for viscosity reduction can be added to the plastic, in particular the plasticized plastic. The diluent is preferably added to the plastic in an amount of at least 5 % by weight, more preferably at least 9 % by weight, based on the total weight of the plastic. The ratio of plastic to diluent is preferably at least 1:4, preferably at least 1:9. By adding the diluent to the plastic, the mobility of polymer chains can be increased at a given temperature, so that heat input into the plastic can be improved during pyrolysis. Furthermore, due to the viscosity reduction, the risk of plastic overheating in wall areas of the pyrolysis reactor can be reduced, since it is usually heated by a heating device arranged near an outer wall of the pyrolysis reactor. The risk of coking of the plastic during pyrolysis can also be reduced by reducing the viscosity.

By adding the diluent to the plastic, its viscosity can preferably be reduced by at least 30 %, more preferably by at least 50 %, particularly preferably by at least 80 %, based on

the viscosity of the plastic without diluent under the same measurement conditions, in particular at a temperature in the range from 180 to 240 °C. This can improve the pumpability of the plastic, which can facilitate its processing.

When the diluent is added, the plastic preferably has a temperature of at least 120 °C, more preferably a temperature of 150 to 300 °C, in particular 200 to 300 °C. Alternatively or additionally, the diluent can be heated to a temperature of preferably at least 120 °C, more preferably at least 150 °C, in particular to a temperature of 200 to 300 °C, before being added to the plastic. By increasing the temperature of the plastic and/or the diluent, the diluent can be mixed into the plastic more quickly and efficiently. The subsequent pyrolysis can also be carried out more energy-efficiently and faster.

The diluent can be added to the plastic by means of a feed device. The feed device may have a metering device, such as a metering pump. For example, the plastic, in particular the plasticized plastic, can be fed to a mixer, e.g. a static mixer, and mixed there with the diluent. If the plastic is plasticized in an extruder, the diluent can be added directly in the extruder. For this purpose, the feed device can be arranged, for example, in the compression zone or mixing zone of the extruder.

The diluent may comprise a hydrocarbon selected from an alkane, a cycloalkane, and/or an aromatic. As a result of the pyrolysis, such a diluent can be converted into a gaseous and/or liquid product, which can be at least partially separated from the pyrolysis residue and reused. In particular, the diluent may comprise a fraction obtained from crude oil, preferably a heavy oil. The heavy oil can be an oil obtained from petroleum in a petroleum refinery, e.g. a residual oil from a pyrolysis system. The diluent preferably comprises at least a portion of a liquid fraction of the pyrolysis residue. This can be separated, for example, in a hydrocyclone.

The diluent preferably has a boiling temperature (or a lower end of a boiling range) of at least 300 °C, in particular at least 350 °C. As a result, the diluent can be prevented from evaporating immediately after a mixture of plastic and diluent has been introduced into the pyrolysis reactor, but evaporation,

cleavage and/or depolymerization of the diluent can only take place with the progressive residence time of the mixture in the pyrolysis reactor and the associated heating of the mixture. As a result, a homogeneous pyrolysis product can be obtained.

Apart from the wax and the solid, the pyrolysis residue provided in step (a) of the method may also comprise a liquid fraction. The proportion of the liquid fraction is preferably at most 95 % by weight, more preferably 30 to 95 % by weight, in particular 50 to 70 % by weight, based on the total weight of the pyrolysis residue. As the proportion of the liquid fraction decreases, the rate at which at least a portion of the wax dissolves in the solvent can increase, or a smaller amount of solvent may be added to the pyrolysis residue in order to dissolve the wax at least partially in the solvent. As a result, the separation of at least a portion of the wax from the low-solid mixture can also be facilitated. The use of solvents can also be reduced as a result. Thus, the method can be designed more efficiently.

The pyrolysis residue provided in step (a) of the method may be obtained by pyrolyzing the plastic in a pyrolysis reactor and subsequently increasing a solids concentration of the pyrolysis residue. As a result, the proportion of the liquid fraction of the resulting pyrolysis residue can be reduced to preferably a maximum of 95 % by weight, more preferably to 30 to 95 % by weight, in particular to 50 to 70 % by weight, based on the total weight of the pyrolysis residue. In order to increase the solids concentration, a hydrocyclone may be used, which can be connected downstream of the pyrolysis reactor.

If the pyrolysis residue is obtained by pyrolysis of a plastic, a gaseous fraction can be separated from the pyrolysis residue after the pyrolysis and before step (a) of the method. The separation of the gaseous fraction can take place by means of evaporation, for example in a hydrocyclone, which can be connected downstream of the pyrolysis reactor.

The separation of the gaseous fraction from the pyrolysis residue and the increase in the solids concentration of the pyrolysis residue can be carried out, for example, with a separation device or with a plurality of separation devices connected

in series. Advantageously, both the separation of the gaseous fraction from the pyrolysis residue and the increase in the solids concentration of the pyrolysis residue take place in only one method step by means of a hydrocyclone, which may be connected downstream of the pyrolysis reactor. Such a hydrocyclone is described in WO 2023/036751 A1. By introducing a mixture comprising the pyrolysis residue and the gaseous fraction via an inlet arranged in an upper region of a shell of the hydrocyclone, a vortex flow can be generated in the hydrocyclone, as a result of which the gaseous fraction can be separated from the pyrolysis residue and discharged via an outlet arranged in the upper region of the hydrocyclone (e.g. on its ceiling). The pyrolysis residue can then be discharged in the direction of a bottom of the hydrocyclone due to gravity, wherein a tangential velocity of a vortex flow that forms can continuously increase. As a result, at least a portion of the pyrolysis residue, in particular at least a portion of the pyrolysis residue at least partially comprising the wax and the solid, can be discharged via an outlet arranged in the bottom of the hydrocyclone, while at least a portion of the liquid fraction of the pyrolysis residue passes into an inner container arranged in the hydrocyclone and can be discharged from there via an outlet, e.g. for further use as a diluent. The pyrolysis residue discharged via the outlet disposed in the bottom of the hydrocyclone can then be provided according to step (a) of the method. The hydrocyclone is preferably operated at a temperature in the range from 300 to 450 °C, more preferably from 320 to 420 °C, particularly preferably from 360 to 400 °C.

The pyrolysis residue may be cooled prior to addition of the solvent, either prior to step (a) of the method, or between steps (a) and (b). Cooling can be carried out by means of a cooling unit. The pyrolysis residue is preferably cooled to a temperature of not more than 220 °C, more preferably not more than 200 °C, particularly preferably not more than 180 °C. The cooling of the pyrolysis residue may be necessary in particular if the pyrolysis residue is obtained by immediately preceding processing of a plastic (by pyrolysis and, if necessary, subsequent separation of a gaseous fraction from the pyrolysis residue and/or increasing the solids concentration of the pyrolysis

residue). By cooling the pyrolysis residue, undesired evaporation and/or decomposition of the solvent during addition to the pyrolysis residue can be reduced or completely prevented. The pyrolysis residue is preferably cooled to a temperature of not less than 30 °C, more preferably not less than 80 °C, even more preferably not less than 100 °C, particularly preferably not less than 120 °C. Further cooling below the stated temperature can impair the solubility of the wax in the solvent.

The solvent may be added to the pyrolysis residue by means of an introduction device. The introduction device may have a metering device, such as a metering pump. In order to mix the pyrolysis residue with the solvent in step (b) of the method, the pyrolysis residue may be fed to a container to which the introduction device can be connected. The container can be heatable so that the dissolving of the wax in the solvent can take place at a certain temperature.

The mixing in step (b) of the method preferably takes place at at least 50 °C (i.e. at 50 °C or above), more preferably at at least 80 °C (i.e. at 80 °C or above). A fraction of the wax with a melting point above room temperature (i.e. above 20 to 25 °C) can then be dissolved in the solvent. The mixing in step (b) preferably takes place at a temperature in the range from 50 to 200 °C, more preferably from 100 to 200 °C, even more preferably from 80 to 200 °C, particularly preferably from 80 to 120 °C. As a result, the wax can not only be dissolved to a large extent or completely in the solvent, but it can also be ensured that the solvent does not evaporate due to an excessively high temperature. The wax can dissolve particularly well in the solvent even at a temperature of 100 to 120 °C, while a temperature of 120 to 140 °C can be optimal in terms of procedure.

The mixing in step (b) of the method preferably takes place at a pressure in the range from 1 to 25 bar, in particular from 5 to 16 bar. As a result, the method can be further improved and rapid dissolution of the wax in the solvent can be achieved.

The solvent preferably has a boiling temperature (or boiling range) in the range from 20 to 250 °C, more preferably from 50 to 150 °C, even more preferably from 35 to 130 °C. The lower the boiling temperature of the solvent, the lower the temperature at

which the wax can be brought into a liquid state. Furthermore, as the boiling temperature of the solvent decreases, the ability of the wax to crystallize during the subsequent cooling of the mixture in step (c) of the method may increase, as a result of which the yield of wax obtained may be increased. The boiling temperature (or boiling range) of the solvent can be determined with the standard ASTM D5399-09:2017 or ASTM D2887-22:2022.

The solvent preferably comprises an aliphatic hydrocarbon or a mixture of two or more aliphatic hydrocarbons. An impurity that dissolves in the solvent can then remain dissolved while the wax is already crystallizing. Thus, the wax can be separated in a high purity and the impurity can remain in the mixture. The impurity may include an organic impurity comprising nitrogen, oxygen, sulfur, silicon, chlorine, bromine, and/or iodine, for example, a heteropolymer (e.g., a polyamide, a polyethylene terephthalate, a polyvinyl chloride, and/or an acrylonitrile-butadiene-styrene copolymer) and/or an additive (e.g., an anti-aging agent, a plasticizer, a color pigment, and/or a flame retardant).

The solvent preferably comprises at least 10 % by weight, preferably at least 20 % by weight, more preferably at least 50 % by weight, of the aliphatic hydrocarbon, or a mixture of two or more aliphatic hydrocarbons, based on the total weight of the solvent. As the proportion of aliphatic hydrocarbon increases, not only can the solvent's ability to dissolve the wax improve, but the solubility of the solid in the solvent can also be reduced. In the case of a high proportion of an aromatic in the solvent, on the other hand, the solid, in particular an asphaltene or a tar, an asphaltene or a tar, can dissolve well in the solvent, which can make the subsequent separation of the wax more difficult, or as a result of which the separated wax can be contaminated. With a high proportion of a cycloalkane in the solvent, the wax can dissolve well, but the yield can be comparatively low.

The aliphatic hydrocarbon is preferably selected from the group of aliphatic hydrocarbons having up to 15 carbon atoms per molecule, in particular from 4 to 12 carbon atoms per molecule. As a result, the method can be carried out efficiently, and good separation of the wax from the low-solid mixture can be made

possible.

The aliphatic hydrocarbon is preferably selected from n-pentane, n-hexane, n-heptane, n-octane, an isomer thereof, or a mixture of the foregoing (i.e., from a mixture comprising n-pentane, n-hexane, n-heptane, n-octane, an isomer of these alkanes, and/or multiple isomers of these alkanes). Since the boiling temperature of these aliphatic hydrocarbons is in the range from 35 to 130 °C, the method can be carried out efficiently and economically. The mixing in step (b) of the method can then preferably take place at 120 °C or below, more preferably at 100 °C or below, in order to avoid evaporation of the solvent. Furthermore, due to the low molecular weight of these solvents, the wax can not only dissolve well in them, but can also have a good tendency to crystallize during subsequent cooling in step (c) of the method.

The solvent preferably comprises at least 10 % by weight of an alcohol, more preferably at least 20 % by weight, based on the total weight of the solvent. As a result, a polar impurity can be dissolved in the solvent and thereby separated from the wax, which crystallizes when the mixture cools. This can further increase the purity of the wax obtained. The solvent preferably comprises 10 to 30 % by weight of the alcohol, more preferably 10 to 20 % by weight of the alcohol, based on the total weight of the solvent. Particularly preferably, the solvent comprises at least 50 % by weight of the aliphatic hydrocarbon and 10 to 30 % by weight of the alcohol. As a result, both the non-polar wax and a polar impurity can be dissolved well in the solvent and separated well from one another during the subsequent cooling of the mixture. Overall, a wax with a high purity can then be obtained.

The alcohol may preferably be selected from methanol, ethanol, propanol, or a mixture thereof. Propanol is particularly preferred because this can reduce adhesions between wax crystals. This may allow easier separation of the wax from the solvent in step (d) of the method. Furthermore, after washing, solvents remaining in the wax can subsequently be separated well from the wax during drying of the wax.

The ratio of pyrolysis residue to solvent in the mixture

obtained in step (b) of the method may depend on the melting temperature of the wax. The higher the melting temperature of the wax, the more solvent may be required to dissolve the wax. It is preferred if the ratio of pyrolysis residue to solvent is in the range from 5:1 to 1:5, preferably from 2:1 to 3:1, in particular 1:1. Then the wax can dissolve to a large extent or even completely in the solvent.

If the pyrolysis residue contains a solid, it can be at least partially separated from the mixture before step (c). Separating the solid may include filtration, adsorption, and/or centrifugation. Preferably, the solid is separated by means of adsorption. An adsorbent may be added to the pyrolysis residue, which may comprise an activated carbon and/or a bleaching earth. As a result, the solid can be separated from the mixture efficiently and as completely as possible.

The solid may comprise an inorganic salt, a ceramic raw material, an asphaltene, a tar, and/or a coke. In particular, the solid may comprise talc, an iron oxide (e.g., ferric oxide), aluminum oxide, titanium dioxide, magnesium oxide, and/or calcium carbonate. If the provided pyrolysis residue is obtained by pyrolyzing a plastic, the solid may comprise an additive contained in the plastic. For example, the additive may comprise a filler, a color pigment, and/or an additive. A specialist knows which additives are used depending on the respective plastic and field of application.

The solid can be dried after it has been separated from the mixture, for example in an oven. As a result, the solid can be made free-flowing and thus easier to process. The solid is preferably dried at a temperature in the range from 50 to 250 °C, more preferably from 100 to 200 °C, even more preferably from 130 to 160 °C. The drying time is preferably up to 120 min, in particular 5 to 60 min.

One or more components can be separated from the solid, in particular from the dried solid, e.g. by filtration and/or centrifugation. Separated components can then be reused, e.g. as an additive for a plastic.

Solvent separated during drying of the solid may be reused in step (b) of the method. Before being recycled into the

method, the solvent can be purified, preferably by evaporation, in particular by rotary evaporation.

In step (c) of the method, the mixture is cooled to crystallize at least a portion of the wax dissolved in the solvent. The mixture is preferably cooled at a rate of at most 25 °C/min, more preferably at most 15 °C/min, more preferably at most 10 °C/min, more preferably at most 5 °C/min, more preferably at most 2 °C/min. Cooling preferably takes place at a rate in the range from 0.05 to 25 °C/min, more preferably from 0.1 to 15 °C/min, more preferably from 0.2 to 10 °C/min, more preferably from 0.4 to 5 °C/min, more preferably from 0.5 to 2 °C/min. Wax crystals can form well in this area at a slow cooling rate, which may allow for better mechanical separation. It has also been shown that slow cooling can lead to a clean product. Faster cooling, on the other hand, can lead to an increased formation of microcrystals, which are harder to separate mechanically and which can also carry more impurities with them.

It is therefore preferred if the cooling in step (c) of the method takes place over a period of at least 5 min, more preferably at least 10 min, more preferably at least 30 min, more preferably at least 60 min. Cooling preferably takes place over a period of 5 to 240 min, more preferably from 10 to 180 min, more preferably from 30 to 150 min, more preferably from 60 to 120 min. The wax crystals that form can have a scale-like shape that is characteristic of these method parameters.

In step (c) of the method, the mixture is preferably cooled to 10 °C or below, more preferably to 0 °C or below, even more preferably to -10 °C or below. Although the wax can in principle already crystallize during cooling at a slow cooling rate, in particular a maximum of 5 °C/min, from, for example, 100 to only 50 °C, a relatively large amount of solvent can be enclosed in the wax crystals, which can reduce the purity of the wax, especially since the solvent can also contain an impurity dissolved therein, which can then also be enclosed in the wax crystals. When cooled to a lower temperature (e.g. to 10 °C or below), the inclusion of solvent can be reduced or completely prevented, so that the purity of the wax obtained can be significantly increased.

In step (d) of the method, at least a portion of the crystallized wax may be separated from the mixture. The separation may comprise filtration and/or centrifugation. The temperature during separation is preferably 80 °C or below, more preferably 30 °C or below. The temperature is preferably in the range from -10 to 80 °C, in particular from 0 to 30 °C. This can prevent the wax from dissolving again in the solvent. Furthermore, the separation can preferably take place at a pressure of up to 10 bar, more preferably at 5 to 10 bar. This can improve method efficiency.

After separating the wax from the mixture in step (d) of the method, solvent contained in the mixture can be at least partially separated from the mixture. The separation can be carried out by evaporation or distillation. Separated solvent may be re-used in step (b) of the method. Before being recycled into the method, the solvent can additionally be purified, preferably by evaporation, in particular by rotary evaporation.

The wax separated in step (d) of the method may have a boiling temperature (or a lower end of a boiling range) of preferably at least 270 °C, more preferably at least 300 °C, even more preferably a boiling temperature (or a boiling range) in the range from 340 to 700 °C. The boiling temperature (or the boiling range) of the wax can be determined by means of the standard ASTM D7500-15:2019 (preferably for a wax with a boiling temperature or a boiling range of 100 to 850 °C, in particular 100 to 735 °C) or by means of the standard ASTM D2887-22:2022 (preferably for a wax with a boiling temperature or a boiling range of 55 to 538 °C) .

The separated wax preferably has at least 15 carbon atoms per molecule, more preferably at least 20, in particular at least 40. The wax preferably has 20 to 80 carbon atoms per molecule, in particular 20 to 65. Such waxes are well suited for further use.

The separated wax can be supplied to a processing plant of a refinery, in particular a fluid catalytic cracking (FCC) plant, a thermal gasoil unit (TGU plant), a hydrogenation plant and/or a coker. The separated wax can also be used in other technical areas, for example as a lubricant and/or additive. The separated

wax can be reprocessed before further use. For example, the wax may be purified and/or separated into different carbon fractions.

The separated wax preferably has a proportion of one (or more) carbon fraction of at least 60 % by weight, more preferably at least 70 % by weight, even more preferably at least 80 % by weight, in particular at least 85 % by weight, based on the total weight of the separated wax. This proportion provides information about the purity of the separated wax, whereby a high proportion of the carbon fraction corresponds to a high purity of the separated wax. By means of the method according to the invention, a wax with a high purity can thus be obtained, which can be increased even further by a subsequent washing of the wax. The proportion of the carbon fraction can be determined by means of a gravimetric analysis, wherein the gravimetric analysis can comprise a determination of the masses of the pyrolysis residue, the added solvent and the separated wax.

The separated wax can be washed after step (d) of the method. In particular, the wax can be washed one to three times. As a result, the purity of the separated wax can be further increased.

During washing, the temperature of the wax is preferably 80 °C or below, more preferably 30 °C or below, even more preferably in the range from -10 to 80 °C, in particular from 0 to 30 °C. This can prevent the wax from dissolving again in the solvent. When washing the wax, a similar or the same temperature can be selected as when separating the wax in step (d) of the method.

Preferably, the wax is washed with an alcohol. As a result, a polar impurity that is still present can be washed out, whereby a wax with a particularly high purity can be obtained. The alcohol used for washing is preferably selected from methanol, ethanol, propanol, or a mixture thereof. As a result, not only can a polar impurity still present be washed out well, but solvent remaining in the wax after washing can subsequently be separated well from the wax during drying of the wax.

The method may comprise a further step (e): drying the separated wax. As a result, solvent separated together with the wax

crystals can be removed. Drying can be carried out thermally at an elevated temperature, or alternatively at a low temperature under vacuum. The type of drying may depend on the further use of the wax. In order to maintain the structure of the wax crystals, drying under vacuum is preferred. If the wax is subsequently used in liquid form, it can also be dried at an elevated temperature and, if necessary, melted. The thermal drying is preferably carried out at a temperature of 150 °C or below and at ambient pressure (0.7 to 1.1 bar). The drying under vacuum preferably takes place at a temperature of 30 °C or below, and at a pressure of 750 mbar or below, in particular at 500 mbar or below. Solvent separated during drying of the wax may be re-used in step (b) of the method. Before being recycled into the method, the solvent can additionally be purified, preferably by evaporation, in particular by rotary evaporation.

The invention relates in particular to the following embodiments:

1. A method for obtaining a wax from a pyrolysis residue, comprising the steps of

(a) providing the pyrolysis residue, wherein the pyrolysis residue comprises the wax,

(b) mixing the pyrolysis residue with a solvent at at least 30 °C to obtain a mixture, wherein the wax is at least partially dissolved in the solvent,

(c) cooling the mixture to crystallize at least a portion of the dissolved wax,

(d) separating at least a portion of the crystallized wax from the mixture.

2. The method according to embodiment 1, wherein the pyrolysis residue is obtained by pyrolyzing a plastic, in particular a waste plastic.

3. The method according to any of the preceding embodiments, wherein the pyrolysis residue is obtained by pyrolyzing a plastic and separating at least one fraction, preferably a gaseous fraction, from the pyrolyzed plastic.

4. The method according to any of the preceding embodiments, wherein the pyrolysis residue has a boiling temperature of at

least 100 °C, preferably at least 150 °C, more preferably at least 200 °C, more preferably at least 240 °C, more preferably at least 270 °C, more preferably at least 300 °C.

5. The method according to any of the preceding embodiments, wherein the pyrolysis residue has a boiling temperature in the range from 100 °C to 700 °C, preferably from 150 °C to 600 °C, more preferably from 200 °C to 500 °C, more preferably from 240 °C to 460 °C, more preferably from 270 °C to 430 °C, more preferably from 300 °C to 400 °C.

6. The method according to any of the preceding embodiments, wherein the plastic comprises a polyolefin and/or a polystyrene (PS), wherein the polyolefin may comprise a polyethylene (PE) and/or a polypropylene (PP).

7. The method according to embodiment 6, wherein the plastic comprises the polyolefin and/or the polystyrene in an amount of at least 65 % by weight, more preferably at least 70 % by weight, in particular at least 90 % by weight, based on the total weight of the plastic.

8. The method according to any of the preceding embodiments, wherein the plastic has a PE content of at least 10 % by weight, preferably at least 20 % by weight, more preferably at least 30 % by weight, more preferably at least 40 % by weight, more preferably at least 50 % by weight, more preferably at least 60 % by weight, even more preferably at least 70 % by weight.

9. The method according to any of the preceding embodiments, wherein the pyrolysis residue is cooled to a temperature of not more than 220 °C, more preferably not more than 200 °C, particularly preferably not more than 180 °C, before the addition of the solvent.

10. The method according to any of the preceding embodiments, wherein the pyrolysis residue is cooled to a temperature of not less than 30 °C, more preferably not less than 80 °C, even more preferably not less than 100 °C, especially preferably not less than 120 °C, before the addition of the solvent.

11. The method according to any of the preceding embodiments, wherein the mixing in step (b) takes place at least 50 °C, more preferably at least 80 °C, in particular in the range from 50 to

200 °C, preferably from 100 to 200 °C, more preferably from 80 to 200 °C, in particular from 80 to 120 °C.

12. The method according to any of the preceding embodiments, wherein the mixing in step (b) takes place at a pressure of 1 to 25 bar, preferably of 5 to 16 bar.

13. The method according to any of the preceding embodiments, wherein the solvent has a boiling temperature in the range from 20 to 250 °C, preferably from 50 to 150 °C, more preferably from 35 to 130 °C.

14. The method according to any of the preceding embodiments, wherein the solvent comprises an aliphatic hydrocarbon.

15. The method according to any of the preceding embodiments, wherein the solvent comprises at least 10 % by weight, preferably at least 20 % by weight, more preferably at least 50 % by weight, of the aliphatic hydrocarbon, based on the total weight of the solvent.

16. The method according to embodiment 14 or 15, wherein the aliphatic hydrocarbon is selected from the group of aliphatic hydrocarbons having up to 15 carbon atoms per molecule, preferably from 4 to 12 carbon atoms per molecule.

17. The method according to any of embodiments 14 to 16, wherein the aliphatic hydrocarbon is selected from n-pentane, n-hexane, n-heptane, n-octane, an isomer thereof, or a mixture of the foregoing.

18. The method according to any of the preceding embodiments, wherein the solvent comprises at least 10 % by weight of an alcohol, preferably at least 20 % by weight, based on the total weight of the solvent.

19. The method according to embodiment 18, wherein the solvent comprises 10 to 30 % by weight of the alcohol, preferably 10 to 20 % by weight of the alcohol, based on the total weight of the solvent.

20. The method according to any of embodiments 14 to 19, wherein the solvent comprises at least 50 % by weight of the aliphatic hydrocarbon and 10 to 30 % by weight of the alcohol, based on the total weight of the solvent.

21. The method according to any of embodiments 18 to 20, wherein the alcohol is selected from methanol, ethanol, propanol, or a mixture thereof, particularly propanol.

22. The method according to any of the preceding embodiments, wherein the ratio of the pyrolysis residue to the solvent in the mixture obtained in step (b) is in the range from 5:1 to 1:5, preferably from 2:1 to 3:1, in particular 1:1.

23. The method according to any of the preceding embodiments, wherein a solid contained in the pyrolysis residue is at least partially separated from the mixture prior to step (c).

24. The method according to embodiment 23, wherein the solid comprises an inorganic salt, a ceramic raw material, an asphaltene, a tar, and/or a coke.

25. The method according to embodiment 23 or 24, wherein separating the solid comprises filtration, adsorption, and/or centrifugation.

26. The method according to embodiment 25, wherein separating the solid comprises adsorption, wherein an adsorbent is added to the pyrolysis residue, and wherein the adsorbent preferably comprises an activated carbon and/or a bleaching earth.

27. The method according to any of embodiments 23 to 26, wherein the separated solid is dried, preferably at a temperature of 50 to 250 °C, more preferably 100 to 200 °C, and/or for a period of up to 120 min, preferably 5 to 60 min.

28. The method according to any of the preceding embodiments, wherein in step (c), the mixture is cooled to 10 °C or below, preferably to 0 °C or below, more preferably to -10 °C or below.

29. The method according to any of the preceding embodiments, wherein the mixture is cooled in step (c) at a rate of at most 25 °C/min, preferably at most 15 °C/min, more preferably at most 10 °C/min, more preferably at most 5 °C/min, more preferably at most 2 °C/min, preferably in the range from 0.05 to 25 °C/min, more preferably from 0.1 to 15 °C/min, more preferably from 0.2 to 10 °C/min, more preferably from 0.4 to 5 °C/min, more preferably from 0.5 to 2 °C/min.

30. The method according to any of the preceding embodiments, wherein the cooling in step (c) takes place over a period of at

least 5 min, more preferably at least 10 min, more preferably at least 30 min, more preferably at least 60 min, preferably over a period of 5 to 240 min, more preferably from 10 to 180 min, more preferably from 30 to 150 min, more preferably from 60 to 120 min.

31. The method according to any of the preceding embodiments, wherein separating the crystallized wax in step (d) comprises filtration and/or centrifugation.

32. The method according to any of the preceding embodiments, wherein the temperature in step (d) is 80 °C or below, preferably 30 °C or below, more preferably in the range from -10 to 80 °C, in particular from 0 to 30 °C.

33. The method according to any of the preceding embodiments, wherein the solvent is at least partially separated from the mixture after step (d), wherein separated solvent is preferably reused in step (b).

34. The method according to any of the preceding embodiments, wherein the separated wax has a boiling temperature of at least 270 °C, preferably at least 300 °C, more preferably a boiling temperature in the range of 340 to 700 °C.

35. The method according to any of the preceding embodiments, wherein the wax has at least 15 carbon atoms per molecule, preferably at least 20, in particular at least 40.

36. The method according to embodiment 35, wherein the wax has 20 to 80 carbon atoms per molecule, preferably 20 to 65.

37. The method according to any of the preceding embodiments, wherein the separated wax has a proportion of a carbon fraction of at least 60 % by weight, preferably at least 70 % by weight, more preferably at least 80 % by weight, in particular at least 85 % by weight, based on the total weight of the separated wax.

38. The method according to any of the preceding embodiments, wherein the separated wax is washed after step (d), preferably with an alcohol, in particular selected from methanol, ethanol, propanol or a mixture thereof.

39. The method according to any of the preceding embodiments, wherein the temperature during washing of the separated wax is 80 °C or below, preferably 30 °C or below, more preferably in

the range from -10 to 80 °C, in particular from 0 to 30 °C.

40. The method according to any of the preceding embodiments, further comprising step (e): drying the separated wax.

41. The method according to embodiment 40, wherein the wax in step (e) is dried at a temperature of 150 °C or below and a pressure of 0.7 to 1.1 bar.

42. The method according to embodiment 40 or 41, wherein in step (e), the wax is dried at a temperature of 30 °C or below and a pressure of 750 mbar or below, preferably 500 bar or below.

43. The method according to any of embodiments 40 to 42, wherein solvent separated during drying of the wax in step (e) is reused in step (b).

FIG. 1 shows a flow chart of a pyrolysis method wherein wax is obtained from a pyrolysis residue.

As can be seen from FIG. 1, a plastic comprising at least 50 % by weight of a polyolefin is fed to an extruder 1, in which the plastic is plasticized and degassed. The plasticized plastic has a temperature of at least 120 °C and is then added to a static mixer 2. In the static mixer 2, a diluent 3 can be added to the plasticized plastic in order to reduce its viscosity. Alternatively or in addition to the diluent 3, a portion of a liquid fraction 4 separated from a pyrolysis residue may be mixed with the plasticized plastic to reduce its viscosity. The obtained mixture is then fed to a pyrolysis reactor 5, in which the plastic is pyrolyzed at a temperature of 350 to 450 °C. As a result, a pyrolysis product 6 comprising a gaseous fraction and a pyrolysis residue is obtained, the pyrolysis residue comprising a liquid fraction, a wax, and a solid. The pyrolysis product 6 is fed to a hydrocyclone 7 downstream of the pyrolysis reactor 5. First, the gaseous fraction is at least partially separated in the hydrocyclone 7. The separated portion of the gaseous fraction 8 can then be further separated into a light oil (e.g. with a boiling range of 35 to 225 °C) and a heavy oil (e.g. with a boiling range of 225 to 410 °C) (not shown). Furthermore, the liquid fraction is at least partially separated in the hydrocyclone 7. The separated portion of the liquid fraction 4 can be discharged via an outlet 9 of the hydrocyclone 7 and reused in the method for viscosity reduction of the plastic, as described

above. At least a portion of the pyrolysis residue, which at least partially comprises the wax and the solid, is discharged via an outlet 10 arranged in the bottom of the hydrocyclone 7.

As can also be seen in FIG. 1, the pyrolysis residue discharged via the outlet 10 is fed to a first cooling unit 11 for cooling to a temperature in the range from 80 to 240 °C. Subsequently, a solvent 13 comprising at least 20 % by weight of an aliphatic hydrocarbon is added to the pyrolysis residue in a container 12 by means of an introduction device 14 in order to obtain a mixture, wherein the wax is at least partially dissolved in the solvent 13. The aliphatic hydrocarbon is selected from the group of aliphatic hydrocarbons having 4 to 12 carbon atoms per molecule. The mixture obtained has a ratio of pyrolysis residue to solvent in the range from 2:1 to 3:1. The container 12 is heated so that the dissolving can take place at a temperature in the range of 80 to 200 °C. In a separation device 15, the solid is at least partially separated from the mixture by means of adsorption on an activated carbon. The separated solid 16 is then dried in an oven 17 at a temperature in the range from 100 to 200 °C. After the separated solid 16 has dried, individual components can be separated from the solid and reused (not shown). Solvent 13 separated during drying is reused in step (b) of the method by being returned to the container 12 via the introduction device 14. Before being recycled, the solvent 13 may be purified, e.g. by evaporation (not shown). According to FIG. 1, the mixture is then cooled in a second cooling unit 18 at a rate of a maximum of 5 °C/min to -10 °C or below, in order to crystallize at least a portion of the wax dissolved in the solvent. Subsequently, at least a portion of the crystallized wax is separated from the mixture by means of a filter 19. Solvent 13 contained in the mixture is separated in an evaporator 20 and likewise returned to the container 12 via the introduction device 14.

As further apparent from FIG. 1, the separated wax 21 is washed in a washer 22 using an alcohol for washing. The washed, separated wax 21 is then fed to a dryer 23 to dry the wax crystals. The obtained wax 24 can then be put to further use (not shown). Solvent 13 separated during the drying of the separated wax 21 is also returned to the container 12 via the introduction

device 14. Before being recycled, the solvent 13 may be purified, e.g. by evaporation (not shown).

Claims:

1. A method for obtaining a wax from a pyrolysis residue, comprising the steps of
 - (a) providing the pyrolysis residue, wherein the pyrolysis residue comprises the wax,
 - (b) mixing the pyrolysis residue with a solvent at at least 30 °C to obtain a mixture, wherein the wax is at least partially dissolved in the solvent,
 - (c) cooling the mixture to crystallize at least a portion of the dissolved wax,
 - (d) separating at least a portion of the crystallized wax from the mixture.
2. The method according to claim 1, wherein the mixing in step (b) takes place at at least 80 °C.
3. The method according to claim 1 or 2, wherein the solvent has a boiling temperature in the range of 35 to 130 °C.
4. The method according to any one of claims 1 to 3, wherein the solvent comprises at least 50 % by weight of an aliphatic hydrocarbon.
5. The method according to claim 4, wherein the aliphatic hydrocarbon is selected from n-pentane, n-hexane, n-heptane, n-octane, an isomer thereof, or a mixture of the foregoing.
6. The method according to any one of claims 1 to 5, wherein the solvent comprises at least 10 % by weight of an alcohol.
7. The method according to claim 6, wherein the alcohol is propanol.

8. The method according to any one of claims 1 to 7, wherein a solid contained in the pyrolysis residue is at least partially separated from the mixture prior to step (c), preferably wherein separating the solid comprises an adsorption, wherein an adsorbent is added to the pyrolysis residue, and wherein the adsorbent preferably comprises an activated carbon and/or a bleaching earth.

9. The method according to any one of claims 1 to 8, wherein the mixture is cooled in step (c) at a rate of at most 5 °C/min.

10. The method according to any one of claims 1 to 9, wherein the mixture is cooled in step (c) to -10 °C or below.

11. The method according to any one of claims 1 to 10, wherein separating the crystallized wax in step (d) comprises filtration and/or centrifugation.

12. The method according to any one of claims 1 to 11, wherein the temperature in step (d) is 80 °C or below.

13. The method according to any one of claims 1 to 12, wherein the separated wax has a boiling temperature of at least 270 °C.

14. The method according to any one of claims 1 to 13, further comprising step (e): drying the separated wax.

15. The method according to any one of claims 1 to 14, wherein solvent separated during drying of the wax in step (e) is reused in step (b).