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STEAM GASIFICATION OF MULTIWIRE LIYCY TYPE ELECTRICAL CABLE

ZGAZOWANIE PARĄ WODNĄ WIEŁOŻYŁOWEGO KABLA ELEKTRYCZNEGO TYPU LIYCY

Abstract: Original, non-separated pieces of the LIYCY cable (with multilayer metal/plastic (copper/PVC) structure) were gasified by steam in an excess at atmospheric pressure. Conversion of the gaseous stream was enhanced by catalytic bed of original granulated material, prepared from aluminosilicate (local clay) and calcium carbonate. In the process metal (Cu) preserved unchanged form of cords and braids and was quantitatively separated (49 % of original mass of the cable). Non-metal components (51 % of original mass of the cable) were converted to a slightly sintered non-metallic powder (3.3 % of original mass of the cable) and gaseous phase. Condensation of steam facilitated elimination of tars and oils as well as hydrochloride from the gas. It was estimated that only 5 % of carbon (from the cable components) was retained in the cooling/condensing line, mostly as water non-soluble phases. Efficiency of absorption of hydrochloride by catalytic bed and aqueous condensate was almost the same (but only 50 % of estimated total chlorine quantity was finally balanced).

Keywords: waste, electrical cables, pyrolysis, gasification

Introduction

Processing of electrical cable and wire waste is usually set to recover of metals, in the first place of copper, although recycling of some polymers has been also developed [1]. The process involves numerous mechanical operations, such as grinding, size classification, separation according to physical properties etc. Recovered metal is recycled as a copper scrap. Multiwire flexible cables represent a significant part of the electronic waste stream, which is processed especially due to high content of precious metals and copper. Recycling of electronic waste is extensively discussed in the literature, with a special attention to thermal processes [2–7]. In dedicated technologies,

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processing of these waste is carried out in reactors with molten metal bath with firing of plastics in oxygen enriched atmospheres; examples are: Noranda process, Kaldor furnace of Boliden or Umicore Isasmelt furnace [3]. In more typical copper pyrometallurgy, the presence of halides is strongly unwanted (however, volatilization of metal chlorides as a method of metal recovery is also considered in the literature [8]). On the other hand, steam gasification of electronic waste was discussed in the literature only for the supercritical state [9, 10]. Multiwire, layered, thin cables with polyvinyl chloride shielding are probably one of the major sources of chlorine in electronic waste processing. Moreover, they are combination of PVC and copper. It was reported in the literature [11] that co-presence of PVC and copper metal results in high emission of chlorinated species under pyrolytic conditions, with dominance of furans over dioxins, as well as high chlorination degree of organic compounds. Gasification with steam enables almost complete elimination of char from metals/inorganic structure and at the same time high concentration of oils and tars (condensing phases) in the product gas may be significantly reduced by catalysts [12].

Laboratory experiment described in this paper was conceived as a high temperature steam gasification (HTSG) processing of original PVC cable waste with the intention of copper components recovery and co-condensation of low-volatile species with excess of the steam in an external cooler.

Experimental section

Gasified material

Gasification experiment was performed with the LiYCY cable cut into four pieces of approximately 10 cm length. The cable was composed of 5 tinned copper multiwire cords (25 wt. %) in a colored polyvinyl chloride (*polwinit*) shielding (17 wt. %). The cords were wrapped with polyester foil (1 wt. %) and screened by the copper braid (24 wt. %). External coating of the cable was also made from polyvinyl chloride (*polwinit*) (32 wt. %).

Catalyst

Equilibration of gaseous phase was promoted by catalytic granules with diameter of approximately 5–10 mm. The height of the catalytic bed was approximately 10 cm. Granules were prepared by mixing calcium carbonate (p.a. POCH S.A.) with clay from local deposit (*Dzierzoniow county*), which was previously dried at ambient temperature and pulverized below 0.25 mm. The mixture was blended with polyethylene glycol (Carl Roth GmbH, ROTH 600, molar mass: 57–630 g/mol) and water, then homogenized. Proportion by weight of clay / carbonate / glycol / water was equal to 21/52/11/16. Catalytic granules were formed from this soft and plastic material and dried in 24 hours at 120 °C, then fired 9 hours at 950 °C and cooled down in several hours to ambient temperature. Weight losses at drying and firing were equal to 16 and 46 %, respectively. Preparation procedure was based on the one given in the literature

[13]. There was similar concentration of Al and Si (9.0 and 11.6 wt. %, respectively) in the final product and high concentration of Ca (21 wt. %). Concentrations of Fe and Mg were equal to 2.2 and 1.4 wt. %, respectively. Specific surface area (S_{BET}) was equal to approximately $16 \text{ m}^2/\text{g}$, the value typical for macroporous solids (with domination of pores with diameter $>0.05 \mu\text{m}$ and clearly smaller share of mesopores with diameter below $0.05 \mu\text{m}$, according to porosimetric analysis).

Reactor and experimental procedure

Gasification experiment has been performed in a quartz reactor which was a simple construction with quartz tubes of different diameters and lengths (Fig. 1).

Gasified sample of the cable pieces was placed on the perforated spacer made of heat-resistant steel (2), supported on the quartz tube with diameter of 22 mm and length of 500 cm (1c). This tube was inserted in the next one (1b) of 30 mm of diameter and 750 cm length, also covered by the perforated spacer (2). This spacer supported the catalytic bed. The third tube (1a), with diameter of 38 mm and 1000 cm length, was the external wall of the reactor. The reactor was sealed at the bottom by water sealing (5) and at the top with the temperature-resistant (up to $285 \text{ }^\circ\text{C}$, Sodual Ltd.) polysiloxane sealing (4). Temperatures of catalytic bed, upper and bottom parts of the sample were measured by three thermocouples K-type (8), placed in the center of the reactor in a quartz tube shielding (3). Temperatures were registered with digital recorder APAR, AR 206/8. Water for generation of steam was supplied by tube (1d) at the bottom of the reactor. Reactor was heated by three tube furnaces (7a, b, c) separately powered by electronic controllers RE31 and RE15 with SSR units (furnaces 7c and 7b, respectively) and autotransformer (7a furnace). Temperatures of the furnaces were controlled by K-type thermocouples (6a, b, c). Polysiloxane sealing (4) was enforced at the bottom by mineral wool layer, and gases flowed from the reactor passing through the glass tube fixed in the

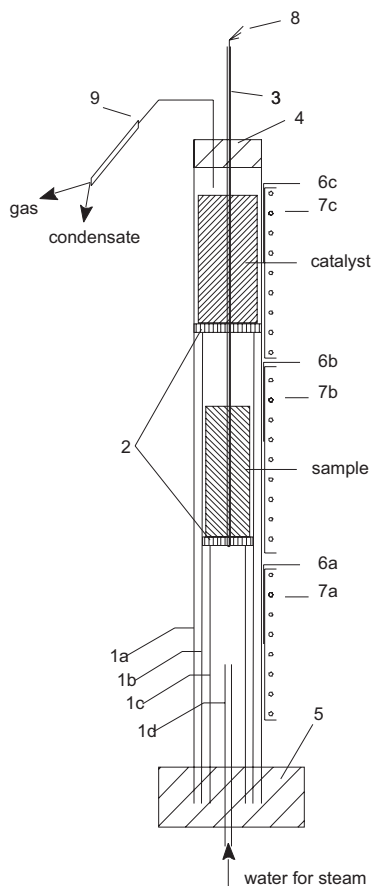


Fig. 1. Reactor used in the experiment: (1) – quartz tubes, (2) – perforated steel spacers, (3) – quartz shielding for thermocouples, (4) – polysiloxane sealing, (5) – bottom water sealing, (6) – thermocouples for controlling of the furnaces, (7) – tube furnaces, (8) – thermocouples for registering temperatures of the sample and catalytic bed, (9) – Liebig condenser

sealing. The tube was fitted to the Liebig condenser (9) with the ground glass joint. Excess of unreacted steam and non-volatile species (tars/oils) condensed in the Liebig condenser (9) and were collected in the glass flask. Non-condensing gases passed out the condensing line through the final water sealing (water flask) and were burned in a micro-flare, if necessary.

Completed reactor (with the sample of the cable and catalyst) as well as condensing line was flushed with argon for 1 hour before the experiment. Then the heating of the catalytic bed and the sample (furnaces 7c and 7b in the Fig. 1) was started. Supply of water for steam generation was started when the temperature of the cable sample was equal to 40–70 °C. The rate of the water supply controlled by peristaltic pump was equal to 0.59 cm³/min, during the whole experiment. The highest temperature of the section with gasified sample was equal to 780 °C. This temperature was kept for 2 hours to almost complete decay of the gas evolution. Finally the reactor was cooled down to the temperatures: 190 °C of the catalytic bed and 125 °C of the gasified material (supply of water was finished when temperature of gasified material was equal to 400 °C). In order to avoid water absorption by the sample and catalyst, reactor was disassembled at this temperature gradient. Precise temperature profiles of the sample and catalytic bed are presented in Fig. 2.

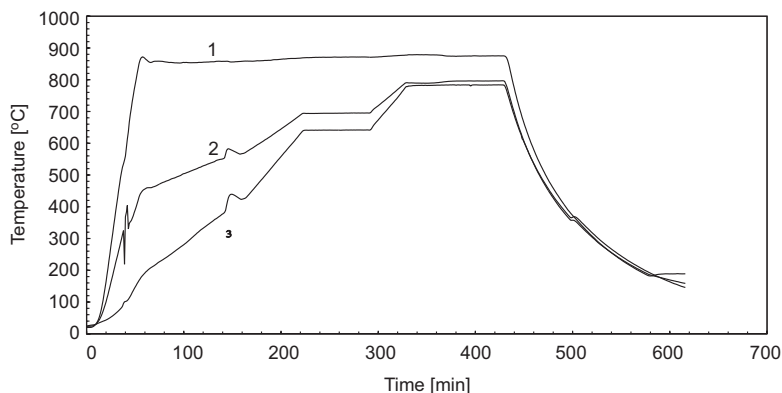


Fig. 2. Temperature profiles of the gasification run: 1 – temperature of the catalyst bed, 2 – temperature of the top of the sample, 3 – temperature of the bottom of the sample

Results and discussion

There were three products of the gasification experiment: (a) non-condensing gases, (b) aqueous condensate, oils and tars and (c) the solid residue in the reactor, dominated by metallic copper.

Non-condensing gases

At the beginning of experiment, reactor and condensing line were flushed by argon. The process started by pyrolysis of PVC, which proceeded, at temperatures above

200 °C, according to the ‘phase chain radical mechanism’ [14]. There were two major degradation steps of (poly)vinyl chloride. During the first step, dehydrochlorination and formation of polyene structures was accompanied by formation of benzene, naphthalene and phenanthrene. In the second phase, there was rearrangement of polyene molecules in the Cl-free melt and cyclization/cross-linking resulting in aromatic hydrocarbons and char residue formation. In a rough temperature scale, dehydrochlorination proceeded between 250–350 °C, formation of polyene structures between 300–450 °C and char/tar formation between 400–500 °C.

Thermal degradation of PVC is a complex process, with a large number of intermediate and final components. Considering presented in the Fig. 2 temperature gradient of the sample during the first 2–3 hours of the experiment, it may be concluded that the process started with dechlorination of the upper part of the sample approximately after 30 minutes from the beginning of the experiment. Then dechlorination proceeded down with successful release of aromatics as well as char formation in the sample sections with the higher temperature. Consequently, gaseous phase which reached catalytic bed was composed of hydrochloride, organic species (probably with a large ratio of aromatics) and steam. With increasing temperature of gasified sample the release of HCl and organic compounds decreased. It may be assumed, that nearly 450–500 °C the pyrolytic decomposition was finished and gasification of the char was started. Intensity of gas emission was noticeably smaller, despite the fact that increasing temperature caused intensification of char transformation process. Hence composition and volume of the gas leaving the reactor and condensing line was steadily changing (and it was qualitatively observed). The main objective of the experiment was to eliminate organic components from the cable and to convert them to non-condensing species, thus the final gas composition was not determined. Moreover, the process in the non-laboratory scale should be continuous and with a specific, stable composition of the

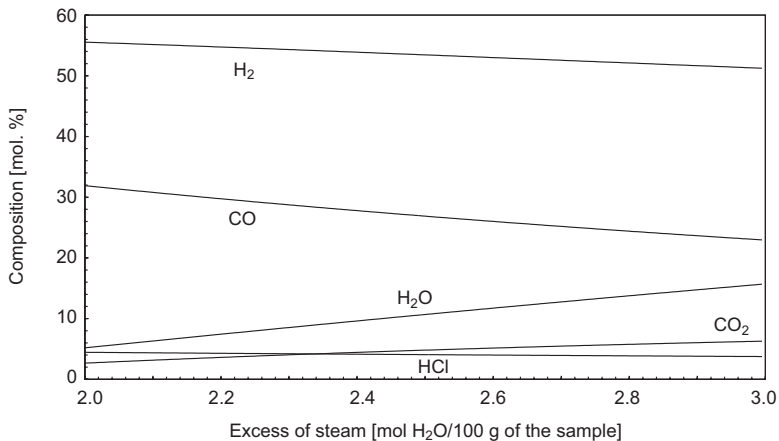


Fig. 3. Major components of the gaseous phase in thermodynamic equilibrium for steam gasification of the cable sample at 850 °C. Calculations have been carried out with HSC Chemistry® for Windows software [15] for an excess of steam (stoichiometric demands are equal approximately to 1.66 moles H₂O/100 g of the sample)

gas, characteristic for the flow reactor. Such calculated gas composition, assuming that the system is in the thermodynamic equilibrium, is presented in Fig. 3 (it should be noted that HCl concentration varies from 3.8 to 4.5 mol. %).

Condensate

Condensation of the steam excess began when temperature of the bottom part of the cable sample exceeded 120 °C. The condensate, colorless and clear at the beginning, became slightly darker and pink with time and separation of small amounts of light oils as well as sediments was observed at the end of experiment (it should be noted that condensing steam washed oils and sediments from the Liebig condenser). The smell of condensate was typical aromatic (naphthalene) and the solution was acidic (pH was approximately 1.0).

Detailed analysis of organic compounds in condensate was performed with mass spectrometry – gaseous chromatography (GC-MS, HP6890/HP5973 and capillary column HP1701 30 m × 0.25 mm with 14%-cyanopropylphenyl-86%-Poly(dimethylsiloxane), He carrier gas, sensitivity of 15–400 u). Four samples of different origins were analyzed: KCa1 – tars/sediments from the container walls, KCa2 – filtered aqueous condensate, KCaS – sediments separated from condensate by filtration and KCaW – tars/sediments from the Liebig condenser. Samples were extracted with diethyl ether (p.a. POCH S.A.) and NIST database was used to identify the species. Results of GC-MS analysis are presented in Table 1.

Table 1

Results of GC-MS analysis of the condensed fractions. KCa1 – tars/sediments from the container walls, KCa2 – filtered aqueous condensate, KCaS – sediments separated from condensate by filtration, KCaW – tars/sediments from the Liebig condenser

No.	Compound	CAS	KCa1	KCa2	KCaS	KCaW
			Mass [mg]			
1	Toluene	000108-88-3	0.12	0.32	0.00	0.06
2	Phenylethyne	000536-74-3	0.04	0.10	0.00	0.03
3	Styrene	000100-42-5	0.26	0.24	0.00	0.16
4	Benzene, 1-ethenyl-2-methyl-	000611-15-4	0.02	0.00	0.00	0.03
5	Indene	000095-13-6	0.45	0.11	0.14	0.74
6	Dodecane	000112-40-3	0.02	0.04	0.02	0.02
7	Naphthalene	000091-20-3	24.72	3.11	10.38	58.79
8	Naphthalene, 2-methyl-	000091-57-6	2.31	0.07	1.18	3.36
9	Naphthalene, 1-methyl-	000090-12-0	1.29	0.04	0.66	1.76
10	Naphthalene, 2-ethenyl-	000827-54-3	4.11	0.08	2.36	5.33
11	Naphthalene, 2,7-dimethyl-	000582-16-1	0.04	0.00	0.02	0.05
12	Naphthalene, 1,7-dimethyl-	000575-37-1	0.13	0.00	0.09	0.16
13	Biphenylene	000259-79-0	0.10	0.00	0.06	0.12
14	Naphthalene, 1-ethenyl-	000827-54-3	0.71	0.00	0.49	0.94
15	Acenaphthylene	000208-96-8	5.44	0.11	3.44	6.99

Table 1 contd.

No.	Compound	CAS	KCa1	KCa2	KCaS	KCaW
			Mass [mg]			
16	Diphenylmethane	000101-81-5	0.07	0.00	0.05	0.09
17	Acenaphthene	000083-32-9	0.06	0.66	0.04	0.07
18	Dibenzofuran	000132-64-9	0.21	0.00	0.15	0.26
19	1,1'-Biphenyl, 3-methyl-	000643-93-6	0.03	0.00	0.00	0.03
20	1H-Phenalene	000203-80-5	0.07	0.00	0.06	0.09
21	Fluorene	000086-73-7	1.75	0.19	1.30	2.07
22	1,1'-Biphenyl, 4-ethenyl-	002350-89-2	0.06	0.00	0.06	0.07
23	9H-Fluorene, 2-methyl-	001430-97-3	0.03	0.00	0.00	0.04
24	Dibenzothiophene	000132-65-0	0.07	0.46	0.05	0.06
25	9H-Fluoren-9-one	000486-25-9	0.43	0.13	0.32	0.47
26	Phenanthrene	000085-01-8	11.95	1.10	9.37	13.53
27	Anthracene	000120-12-7	3.57	0.15	3.20	7.68
28	1H-Indene, 1-(phenylmethylene)-	005394-86-5	0.52	0.00	0.42	0.58
29	Anthracene, 1-methyl-	000610-48-0	0.28	0.00	0.21	0.35
30	Anthracene, 2-methyl-	000613-12-7	0.34	0.00	0.26	0.43
31	Phenanthrene, 3-methyl-	000832-71-3	0.12	0.00	0.09	0.19
32	Naphthalene, 2-phenyl-	000612-94-2	1.81	0.00	1.38	2.22
33	1H-Phenalen-1-one	000548-39-0	0.11	0.00	0.06	0.23
34	9,10-Anthracenedione	000084-65-1	0.09	0.00	0.00	0.10
35	Naphthalene, 1-phenyl-	000612-94-2	0.16	0.00	0.08	0.19
36	Pyrene	000129-00-0	5.45	0.22	3.51	6.73
37	Fluoranthene	000206-44-0	2.10	0.11	1.42	2.57
38	Benzo[b]naphtho[2,3-d]furan	000243-42-5	0.24	0.00	0.09	0.16
39	p-Terphenyl	000092-94-4	0.14	0.59	0.00	0.16
40	Triphenylene	000217-59-4	0.10	0.00	0.05	0.00
41	11H-Benzo[b]fluorene	000243-17-4	0.63	0.00	0.36	0.83
42	Pyrene, 2-methyl-	003442-78-2	0.33	0.00	0.18	0.42
43	Pyrene, 1-methyl-	002381-21-7	0.25	0.00	0.13	0.30
44	Benz[a]anthracene	000056-55-3	0.12	0.00	0.06	0.13
45	Cyclopenta[cd]pyrene	027208-37-3	0.35	0.00	0.23	0.52
46	1,2'-Binaphthalene	004325-74-0	0.08	0.00	0.06	0.10
47	Triphenylene	000217-59-4	1.17	0.00	0.69	1.86
48	Chrysene	000218-01-9	1.96	0.51	1.10	3.01
49	7H-Benz[de]anthracen-7-one	000082-05-3	0.12	0.00	0.07	0.18
50	2,2'-Binaphthalene	000612-78-2	0.09	0.00	0.00	0.19
Total			74.62	8.34	43.89	124.45

Total mass of identified species (presented in the Table 1) was equal to 251.3 mg. Fractionation of these species was very characteristic: only 3 % was found in filtered aqueous condensate, 17 % in filtered sediments, 30 % in sediments from the container walls and up to 50 % in sediments from the Liebig condenser. To evaluate the amount

(percentage) of carbon isolated from cable plastic in cooling/condensing line, two parameters were considered: composition of *poliwinit* (discussed later) and average concentration of carbon. The latter was assumed to be equal to 90.0; 38.4 and 73.8 wt %, respectively in compounds listed in Table 1, PVC, and plasticizer (DEHP). Based on these data it was evaluated that 5.3% of carbon was isolated, mostly as water insoluble individuals. Accordingly, 94.7 % of the carbon was converted into non-condensing species.

Solid residue

Gasification experiment was carried out in order to extract metallic copper in the original form (*ie* without melting) and to eliminate the char from metal/inorganic residue. The solid products of gasification are presented in Fig. 4.



Fig. 4. The solid residue of gasification experiment: a) non-metallic residue (with small pieces of copper), b) copper braids, c) cooper cords, d) pieces of cooper manually separated. Copper cords were removed from copper braids after experiment, for better presentation

They are: copper metal in the form of braids (48 % of the metal), cords (52 % of the metal) as well as manually separated little pieces (0.30 % of the metal) and a small amount of gray, slightly sintered powder with slight admixture of tiny pieces of copper. Masses of the original cable sample and fractions of the solid residue of gasification are given in Table 2.

Table 2

Mass balance of gasified sample of the LiYCY cable. The cable was cut into four pieces and placed in the reactor without additional treatment

Unit	Gasified cable	Solid residue	Deficit of mass	Solid residue	
				Non-metal fraction	Metal (Cu)
m [g]	18.0912	9.4692	8.622	0.5991	8.8701
wt. %	100	2.3	47.7	6.3	93.7

Fractionation of chlorine

It was discussed above that decomposition of PVC involves dehydrochlorination as an initial phase of destruction [14]. It seems that there is agreement in the literature that

HCl is the major chlorine product of PVC pyrolysis [16, 17], although temperature range of maximum emission of this compound depends on the material. It was also reported that emission of HCl was accompanied by emission of light hydrocarbons (in the first place benzene) as well as chlorinated light hydrocarbons, with domination of C_3H_5Cl [16]. In the presented here experiment gases from decomposition of PVC were forwarded together with steam to catalytic bed heated to 850 °C. Calculations performed with HSC software [15] revealed that equilibrium concentration of chlorinated organic species is negligible in comparison with hydrochloride. The same calculations show that calcium oxide (in the catalyst granules) should be chlorinated to $CaCl_2$. Additionally, PVC additives (eg calcium carbonate or dolomite in a form of fillers) may also react with hydrochloride [18]. Unfortunately, there was no information on such additives to *polwinit* (PVC in the cable shielding). Therefore, it was assumed that the chlorine from gasified PVC should be principally in the form of HCl and should be captured by: catalytic bed, aqueous condensate and mineral wool, located between catalytic bed and polysiloxane sealing. Analysis of the chloride ions content was performed by Mohr method, after leaching of the wool and the sample of catalyst granules with hot water. Non-soluble sediments from aqueous condensate were separated by filtration. Results of analysis are presented in Table 3.

Table 3

Distribution of chlorine between aqueous condensate, catalytic bed and mineral wool (support of the polysiloxane sealing)

Constituent	pH	Cl ⁻ [mg]
Leaching of the catalyst	~9–10	603
Leaching of the mineral wool	~5–6	23
Aqueous condensate (filtered)	~1	604
Total		1230

There was no available information on *polwinit* composition, therefore it was assumed that the material was composed of 50 % PVC, 40 % plasticizers (in the first place phthalates, eg diethylhexyl phthalate, DEHP), 2–4 % stabilizers (eg metal stearates or carboxylates) and fillers (chalk, talc, dolomite, quartz powder) to the balance. Considering amount of *polwinit* in the cable sample and chlorine content in (poly)vinyl chloride (56.8 wt %), the mass of 1.23 g Cl⁻ corresponds to only 50 % of expected chlorine quantity. Because there was only 0.6 g of non-metallic fraction from the solid residue after gasification, it seems that (1) significant amount of chlorine was not captured by alkaline component of catalytic bed and steam condensation or (2) assumed composition of *polwinit* was not correct. Anyway, the chlorine distribution should be investigated more carefully, taking into account additional alkaline gas-washing and reaction with PVC fillers. Moreover, replacing of calcium oxide by magnesium oxide as catalytic active component should be considered ($p_{H_2O} / p_{HCl}^2 = 2.56 \cdot 10^4$ for the reaction $CaO + 2HCl = CaCl_2 + H_2O$ at 850 °C whereas relevant equilibrium constant for $MgO + 2HCl = MgCl_2 + H_2O$ reaction is equal to $6.79 \cdot 10^{-2}$). Rare earth oxychlorides seems also interesting as possible active components.

Conclusions

It has been shown that multilayer metal/plastic (copper/PVC) structure of the LiYCY cable was efficiently gasified by steam in an excess to metal (Cu) and non-metallic solid residue. Gasification was enhanced by catalytic bed of original granules prepared from local clay and calcium carbonate. In the process, metal (Cu) remained unchanged and was quantitatively separated (49 % of the original mass of the cable). Non-metal components of the cable (51 % of the original mass) were converted to non-metallic solid residue (3.3 % of the original mass of the cable) and gaseous phase. Condensation of steam facilitated elimination of tars and oils as well as hydrochloride from the gas. It was estimated that 95 % of carbon (from the cable components, particularly (poly)vinyl chloride) was converted to non-condensing species and only 5 % of carbon retained in the cooling/condensing line as (mostly) water non-soluble phases. Efficiency of absorption of hydrochloride by catalytic bed and aqueous condensate was almost the same (but only 50 % of estimated chlorine quantity in the sample was finally balanced).

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ZGAZOWANIE PARĄ WODNĄ WIEŁOŻYŁOWEGO KABLA ELEKTRYCZNEGO TYPU LiYCY

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Abstrakt: Oryginał, nie rozdzielone (w całości) kawałki kabla LiYCY (kabel wielożyłowy, kombinacja warstw metal/tworzywo sztuczne (Cu/PCW)) zgazowywano w nadmiarze pary wodnej pod ciśnieniem normalnym. Konwersję strumienia gazów prowadzono na złożu katalitycznym z oryginalnego, granulowanego materiału glinokrzemianowego (lokalna glina), z dodatkiem węgla wapnia. W procesie zgazowania metal (Cu) zachował oryginalną postać linek i opłotów i został ilościowo wydzielony (49 % wag. kabla przed zgazowaniem). Niemetaliczne składniki kabla (51 % wag. kabla przed zgazowaniem) zostały przekształcone do nieznacznie spieczonego, niemetalicznego proszku (3,3 % wag. kabla przed zgazowaniem) i gazu. Kondensacja pary wodnej wspomagała usuwanie z gazu substancji smolistych i olejowych, a także chlorowodoru. Oszacowano, że tylko 5 % węgla (zawartego w tworzywach kabla) zostało zatrzymane w układzie chłodzenia/kondensacji, w zdecydowanej większości jako nierozpuszczalne w wodzie fazy. Skuteczność absorpcji chlorowodoru przez złożo katalityczne i kondensat wodny była praktycznie taka sama (ale zbilansowano ostatecznie tylko 50 % oszacowanej ogólnej zawartości chloru).

Słowa kluczowe: odpady, kable elektryczne, piroliza, zgazowanie

