International Journal of Public Health Science (IJPHS)
Vol. 13, No. 4, December 2024, pp. 1952~1959
ISSN: 2252-8806, DOI: 10.11591/ijphs.v13i4.24391

Holistic characterization analysis of tar waste content from gasification process at Surakarta landfill

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Article Info

Article history:

Received Dec 22, 2023
Revised Mar 21, 2024
Accepted Apr 24, 2024

Keywords:

Acute toxicity
Gasification
Heavy metals
Tar waste
Waste power plant

ABSTRACT

Surakarta is facing serious problems related to waste management at the Putri Cempo landfill. PT. Solo Citra Metro Plasma Power with the Surakarta city government built a waste power plant installation with gasification technology. Tar waste is a gasification liquid product that is dangerous if not managed. This study aimed to determine the heavy metal content and toxicity level of gasified tar waste using the atomic absorption spectrometry (AAS) and lethal dose-50 (LD_{50}) acute toxicity test. The parameters of heavy metals tested are lead (Pb), copper (Cu), iron (Fe), zinc (Zn), and total chromium (Cr). In the LD_{50} test, the animals were divided into 5 groups, namely 1 control group and 4 groups with doses of 0.50 mL, 1 mL, 2 mL, and 4 mL. The results showed that the tar contained 17.4 mg/L of iron, 3.5 mg/L of zinc, and <0.01 mg/L of lead, copper, and chromium. Acute toxicity tests did not cause death in the animals but still showed toxic symptoms, so the LD_{50} value is declared pseudo. The content of some heavy metals in tar is within safe limits and the level of toxicity is relatively harmless.

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1. INTRODUCTION

Surakarta is facing serious problems related to significantly increased waste production. Based on data from the Environment Agency of Surakarta in 2021, the people of this city produced 5,873,750 m^3 of waste every day. The only dumping site is Putri Cempo Final Processing Site, with a capacity of around 3,000,000 m^3. Because the disposal mechanism still uses the open dumping method, community waste accumulates to exceed the capacity of the landfill itself [1]. In response to this issue, the government took the initiative to develop waste processing in the form of electrical energy utilization. A waste power plant (WPP) is presented as an answer to waste problems based on the concept of waste to energy [2]. PT. Solo Citra Metro Plasma Power (SCMPP) together with the Surakarta city government built a WPP that is estimated to serve 550 tons of waste every day, which can be converted into 5 MW of electricity.

The WPP operates primarily on the principle of gasification, a thermochemical process that converts energy from solid fuels through incomplete combustion. In Indonesia, gasification as a waste management method is yet to be widely adopted due to its high cost and substantial feedstock requirements. However, gasification offers several advantages, including its ability to process various types of waste, its ease of development, and its non-production of greenhouse gases [3]. The gasification process yields synthetic gas/syngas (CO, CO_2, CH_4, H_2), char and ash (solid phase), and tar (liquid phase) as output products [4]. While syngas is currently used to power turbine generators and charcoal is utilized as fuel for power plants, the potential of tar still needs to be explored.

Tar is defined as a mixture of various hydrocarbon chains that have a considerable amount of oxygen and water content [5]. This liquid is produced from condensed steam so that there is a phase change from steam to liquid. Research related to tar from waste gasification results is still relatively new and has yet to be widely studied, especially in Indonesia. The same mechanism processes tar from waste gasification and coal gasification, but the type of tar produced has different qualities. Based on the research, the content of carbon and sulfur elements in coal tar is higher than in waste tar [6]. Waste tar also has a small level of phenol, which has the ability to kill microorganisms [7]. Tar produced from gasification cannot be disposed of directly into the environment because tar has mutagenic and toxic properties. Exposure to these substances in the human body decreases the potential of mitochondrial membranes to oxidative damage and can even cause death in body cells [8]. This problem is the main reason for carrying out this study, which aims to determine the heavy metal concentration and toxicity level of the gasified tar waste at PT. SCMPP.

2. METHOD

2.1. Materials

This research was conducted at PT. SCMPP, Surakarta as a sampling site. Tar sampling at PT. SCMPP uses the judgment technique, which is a way of sampling at its discretion at a representative point. The requirements for samples to be tested using the AAS method are pure samples, have a minimum water content of 0.1%, must be separated from physical materials that can be deposited or cannot be precipitated, and the concentration of metals that can be determined must be above the minimum detection limit. Tar samples are taken in special concrete tar reservoirs with metal covers protected from the ingress of other liquids.

2.2. Heavy metal content

Atomic absorption spectrometry (AAS) is an industry-standard for heavy metal content assessment due to its high sensitivity, ease of use, simple configuration, and somewhat low operating costs [9]. Using AAS instruments is also quite straightforward because metal components do not need to be separated from the materials. The working principle of AAS is the absorption of light by elements because each element has the ability to absorb light energy at a certain wavelength, depending on its nature. This assessment is carried out in accordance with SNI 6989.84:2019 about how to test dissolved and total metal levels by AAS–Flame. Prior to testing, the sample will be destroyed with HNO3 after being filtered using filtration media having pore sizes of 0.45 µm. Subsequently, the primary metal solution will be diluted to create 100 mL of metal working solution and 10 mL of metal raw solution. The working solution and blank solution will be inhaled through the aspirator hose once the AAS has been turned on and optimized. By heating the sample to a high temperature, the sample will become free atoms. The atomized sample will be exposed to light from the metal cathode lamp, and the atoms within will absorb light at wavelengths determined by their characteristics. A comparison is made between the intensity of light absorbed and the intensity of light flowing through an empty sample. The information about the elemental concentration in the sample that is shown by the AAS software is provided by this intensity difference. The parameters of heavy metals tested are lead (Pb), copper (Cu), iron (Fe), zinc (Zn), and total chromium (Cr). Metal content is calculated using a formula that complies with SNI 6989.84:2019 (1).

$$Metal\ content\ (mg/L) = C\ x\ fp \quad (1)$$

With the description of the formula:
C : metal content obtained from the measurement results (mg/L)
fp : dilution factor

After obtaining the results of calculating the metal content in tar, the results are adjusted to the wastewater quality standards at hazardous and toxic waste storage facilities contained in the regulation of the Minister of Environment and Forestry number 6 of 2021 concerning procedures and requirements for hazardous and toxic waste management as shown in Table 1.

Table 1. Wastewater quality standards at hazardous and toxic waste storage facilities

No	Parameters	Maximum concentration
1	Iron (Fe)	5 mg/L
2	Copper (Cu)	2 mg/L
3	Zinc (Zn)	5 mg/L
4	Chromium (Cr)	0.5 mg/L
5	Lead (Pb)	0.1 mg/L

2.3. Acute toxicity levels (LD_{50})

The acute toxicity test employed in this investigation was an LD_{50}-based test. This method works by grouping test animals and administering chemicals or compounds in a single dose that is stratified or changes according to the group. Following that, the test animals' behavior will be monitored for alterations for a predetermined amount of time. The LD_{50} value of the test will be established by the quantity of test animal deaths that occur. After 7 and 14 days of dosage, observations were made regarding the state of the test animals' organs in addition to behavior and the number of deaths.

This study used fifteen male BALb/C strain mice who were two months old. The animals were divided into 5 groups, namely 1 control group and 4 groups with doses of 0.50 mL (±0,02 mL/g BB), 1 mL (±0.04 mL/g BB), 2 mL (±0.08 mL/g BB), and 4 mL (±0.16 mL/g BB). Before the tar is given to the animals, the tar will be dissolved in 10% v/v (volume percent) distilled water until a total solution of 30 mL is formed. The material is dissolved with a magnetic stirrer. The purpose of dissolving the tar is to simplify the process of administering the preparation to the animals, especially in this study using the oral method, so it is necessary to ensure that the preparation can flow through the probe properly. Observation of toxic symptoms is carried out for 3-4 hours after the application of tar to animals. After 24 hours, the death of test animals is observed. Observations were made up to 14 days further to see the reversibility of toxic effects. In addition, observations were made on the organs of mice, and the condition of the organs of each group was compared. The Thompson and Weil formula is used to get the LD_{50} value (2).

$$\log m = \log D + d(f + 1) \tag{2}$$

With the description of the formula:
m : LD_{50}
D : smallest dose used
d : log r (multiple of dose)
f : factor

Based on Government Regulation No. 74 of 2001 concerning the Management of Hazardous and Toxic Substances, there are 6 levels of toxicity for LD_{50} testing. The lowest level is relatively harmless (>15.000 mg/kg), practically non-toxic (5.001-15.000 mg/kg), slightly toxic (501-5000 mg/kg), moderately toxic (51-500 mg/kg), highly toxic (1-50 mg/kg), and the highest level is extremely toxic (<1 mg/kg).

3. RESULTS AND DISCUSSION

3.1. Heavy metal content of gasified tar at PT. SCMPP

The parameters of heavy metals tested were Pb, Cu, Fe, Zn, and Cr. The selection of parameters is based on differences in metal groups, where Pb and Cr are classified as highly toxic metals, Cu as moderately toxic metals, and Fe and Zn are classified as fewer toxic metals. Cu, Pb, Cr, and Zn are quite often found in polluted water, and this poses a threat to public health because of the high potential for accumulation in the food chain [10]. The human body also needs metals, such as iron, for the formation of red blood cells. However, excess of these substances can cause infection and increase susceptibility to the risk of cancer and heart attack [11]. The content of these metals in tar will affect the mechanism of management and utilization of the waste, and how it impacts living things and the environment can be identified. Table 2 shows the results of the AAS test of PT. SCMPP gasification tar, which is compared with the 'Quality standards for wastewater at hazardous waste storage facilities in the form of waste piles and waste impoundments in the Regulation of the Minister of Environment and Forestry Number 6 of 2021.

The migration or transport of heavy metals in gasifiers during combustion has been the subject of several investigations. According to a study, when trash is heated, the heavy metals will evaporate or turn into volatile metal particles, which will be taken away as fly ash or bottom ash [12]. High-boiling-point heavy metals like Cr and Cu will settle in the bottom ash, but extremely volatile heavy metals like Cd will leave the combustion zone as exhaust gases [13]. Based on a research, most heavy metals are concentrated in ash, and their content are much higher than those in soil or tar, as another gasification output [14]. In the gasification process at PT. SCMPP, tar is removed from the heat exchanger after the pyrolysis gas is filtered in cyclones and high-temperature element filter, so the air produced is completely clean.

Based on several studies, early signs of iron poisoning include nausea, vomiting, diarrhea, abdominal discomfort, bleeding in the digestive tract, changes in stool color, and hypovolemia, which is a sharp drop in blood or other bodily fluid levels [15]. These symptoms usually appear during the first six hours of the condition. Numerous studies indicate that iron accumulation can reduce bone mineral density and increase the risk of osteoporosis [16]-[18]. Because the human body is unable to eliminate iron, excessive blood transfusions can cause an individual's skin to become black from iron accumulation [19].

Table 2. The result of the heavy metal content of tar PT. SCMPP

No.	Parameter	Unit	Score	Max. concentration	Status
1	Lead (Pb)	mg/L	<0.0099	0.1	Doesn't exceed
2	Copper (Cu)	mg/L	0.0069	2	Doesn't exceed
3	Iron (Fe)	mg/L	17.3700	5	Exceed
4	Zinc (Zn)	mg/L	3.4940	5	Doesn't exceed
5	Chromium (Cr)	mg/L	<0.0095	0.5	Doesn't exceed

The other most abundant heavy metal in tar is Zn. If Zn is consumed 2 grams or more, the body can experience vomiting, diarrhea, anemia, fever, chronic fatigue, and reproductive disorders [20]. The main indicator of whether zinc is harmful or not is how the metal is metabolized within the organism. One of the most frequent causes of acute zinc toxicity is overconsumption of zinc dietary supplements, which leads to an imbalance in copper availability and eventually ends in zinc insufficiency [21]. Excessive zinc consumption may cause abdominal discomfort, diarrhea, nausea, and vomiting [22].

Gasified tar of PT. SCMPP has very little content of Pb, Cu, and Cr, but heavy metals still can accumulate easily in living things bodies and the environment. Pb can cause chronic toxicity when it enters the body because the blood absorbs it and builds up in soft tissues and bones [23]. If the human body is exposed to Pb more than 50 μg/dL, it can inhibit the production of hemoglobin in erythrocytes [24]. In the human adult body, there are 100-150 mg of Cu, of which 10% comes from the liver [25]. Research states that consuming drinking water with copper levels of more than 6 mg/L will cause digestive disorders such as nausea, vomiting, and diarrhea [26].

The amount and composition of tar will vary depending on the raw material and operational conditions during the gasification process, and different reactors can produce different amounts of tar. The source of heavy metals in landfills depends on the composition of the waste. Waste degradation may reduce landfills, but heavy metal concentrations will not decrease; they are redistributed in the leaching process. The distribution of these heavy metals is influenced not only by the chemical and physical properties of waste as inputs but also by operational factors like temperature, gas flow, humidity levels, and gas processing methods.

3.2. Toxicity levels of gasified tar at PT. SCMPP

Toxicity tests aim to measure the degree of toxicity of a substance over a certain time. This research used an acute toxicity test with the LD_{50} method. The working mechanism of this method is that the experimental animals will be grouped, and the substance will be administered in a single dose that varies based on the group. After that, the animals will be observed for changes in their behavior over a certain time. If a death occurs in an animal, the number of deaths will determine the LD_{50} value. Apart from observing the behavior and number of deaths, observations were also made on the condition of the experimental animals body organs after 7 and 14 days of dosing.

The LD_{50} test was carried out using mice (*Mus musculus*) with a 2-month-old BALb/C male type. Male mice are more widely used in research than female mice; this is because the hormonal conditions in female mice are unstable. This hormonal cycle has the potential to cause higher levels of stress compared to male mice. BALb/C is part of strain, a genetic grouping of members through lineages. This type of mouse is characterized by white fur (albino), having a tail that is longer than its body length and has stronger physiological characteristics. BALb/C is one of the strains that is often studied in drug trials and other health research, such as research on cancer and immunology [27].

Fifteen test animals were employed in this investigation; they were split up into five treatment groups, one control group, and four groups with varying dose volumes. Before treatment, the test animals went through an acclimatization period of approximately seven days. Acclimatization is important to ensure that the condition of the test animals is free from discomfort or stress due to changes in the environment [28]. Test animals were weighed before administering the preparation as an initial stage of observing growth and development and toxic effects on mice for 14 days. The distribution of groups of test animals is carried out randomly, where each group will be placed in a different cage. Feeding, drinking, and observation of mice activity are carried out every day.

Tar preparations were given to test animals in groups II-V, with a dose volume of 0.5 mL or equivalent to ±0.02 mL/g BW for group II, 1 ml preparation or equivalent to ±0.04 mL/g BW for group III, 2 ml preparation or equivalent to ±0.08 mL/g BW for group IV, and 4 ml preparation or equivalent to ±0.16 mL/g BW. After administering the preparation, observations will be made whether there is death or not after 3-4 hours, after 24 hours, and up to 14 days after administering the preparation.

3.2.1. Acute toxicity test (LD_{50}) results

Even with the highest dosage, no one dead after receiving tar preparation for 14 days, as seen in Table 3. The LD_{50} value cannot be determined in the absence of test animal mortality because the f factor from the Thomson and Weil biometric table cannot be acquired. According to experts, the LD_{50} is considered false or does not represent the true LD_{50} number if the test animals do not die at the maximum dosage administered [29]. If the test animals get a higher dose of the preparation than what was used in this study, the true LD_{50} can be determined.

The maximum dosage of the preparation given to the test animals is used to calculate the LD50 value because the test animals did not die from the maximum dose used in the experiment. Thus, the highest dose from the acute toxicity test that was carried out was 0.16 mL/g BW or 160,000 mg/kg BW. Based on Government Regulation Number 74 of 2001 concerning Management of Hazardous and Toxic Materials and the Loomis toxicity criteria, tar from PT. SCMPP is included in the "relatively harmless" or "non-toxic" category.

Table 3. Results of the number of living and dead mice after giving tar samples of PT. SCMPP

No.	Group	Dose volumes	Number of mice		
			Total	Live	Dead
1	I	Control	3	3	0
2	II	0.5 mL (±0.02 mL/g BB)	3	3	0
3	III	1 mL (±0.04 mL/g BB)	3	3	0
4	IV	2 mL (±0.08 mL/g BB)	3	3	0
5	V	4 mL (±0.16 mL/g BB)	3	3	0

3.2.2. Results of observation of toxic symptoms in test animals

The tar injection significantly impacts the animal's behavior. After the injection of tar, behavioral observations were made 3 times: 3-4 hours, 24 hours, and 14 days later. The test animals did not show any toxic signs, such as vomiting, shaking, seizures, or other medical symptoms, in response to the offered dose of the preparation. The mice's activity levels changed significantly during these 14 days. Several terms describe the activity or behavior of mice in the laboratory, such as moving, grooming, resting, feeding, social, foraging, exploration, drinking, and nest-building. The mice generally displayed all of these behaviors, although at various levels of intensity. Mice are primarily nocturnal animals, although they can be easily awakened during the day [30]. Only a few activities happen in the morning until noon, as mice are more active from dawn till night. Before tar administration, the mice are very active, even during the day. The intensity of moving, social, and exploratory behavior was quite frequent. After the tar was administered, the mice were silent for a while and then moved as usual. The mice with the highest dose remained silent longer than the mice in the other groups. The 3-4 hours after that, the mice activity intensity was slightly less. The next day, the mice's activities were normal; some of them were exploring the cage or sleeping. The 2-7 days after the injection, mice activities in the cage decreased slowly as the mice became accustomed to the cage environment and interacted with fellow mice in the same cage. All the mice were still alive until the 14th day, but some mice had slightly dirty fur due to their social interaction, and some had small wounds from fighting with other mice. Activity during the day was greatly reduced. The social intensity and exploration decreased significantly compared to the previous days. Sound and vibration stimuli woke them up, but they did not always respond by shifting positions.

3.2.3. Organ observation results of test animals

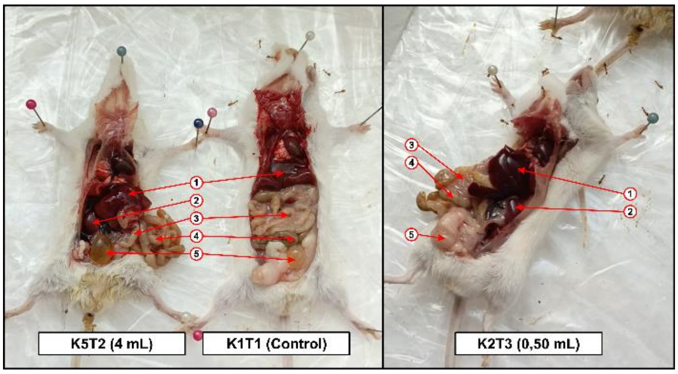
Apart from observing the activity of the mice, observations were also carried out on their internal organs to determine the toxic effects caused by tar PT. SCMPP. On days seven and 14, all mice from each group were separated in order to investigate some of their organs, including the liver, kidneys, stomach, small and large intestines, and bladder. The choice of these organs depends on the oral delivery technique of the preparation, meaning that the treatment's effects may impact the mice's urinary and digestive systems.

On day 7, the mice's internal organs were dissected to determine the short-term effects of tar, especially in the digestive system. The neck dislocation procedure first killed the mice before they were dissected. The mice's tails were held and placed on an accessible surface to complete the operation. The mice lengthened their bodies, and the right hand pulled hard on the tail while the left hand held the nape of the neck, causing the neck to become dislocated and the mice to perish.

At this surgery, 3 mice from different treatment groups were dissected, namely K5T2, K1T1, and K2T3 as shown in Figure 1. The test animals were randomly selected to represent group 1 (control), group 2 (given the lowest dose volume), and group 5 (given the highest dose volume). These three groups represent the different conditions experienced by the test animals. The Figure 1 shows how the organ condition of mice without tar is different from that of mice given tar preparations. K1T1 mice showed a healthy condition; the heart and liver were reddish, there was no swelling in the intestines, and the bladder was still in normal size and brightly colored. This condition is different from K2T3 mice, which began to show signs of swelling in the intestines, and the bladder became slightly enlarged. In K5T2 mice, very significant differences were seen in the heart, which was slightly black, the intestines were swollen, and even the bladder was enlarged and no longer bright in color. Problems in these organs have the potential to be caused by the heavy metal content in the tar from the gasification of PT. SCMPP.

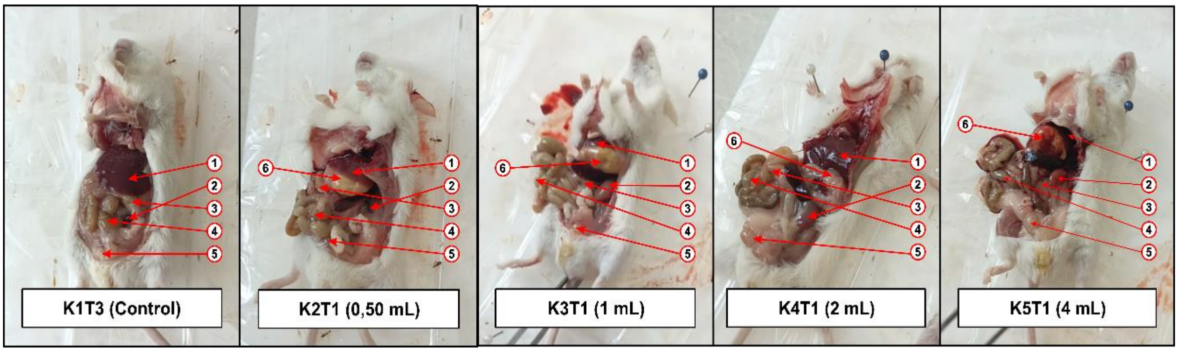
Meanwhile, on the 14th day, surgery was carried out on 8 test animals from each treatment group, namely K1T3, K1T2, K2T1, K3T1, K4T1, K4T2, K4T3, and K5T1. In Figure 2, a comparison of the condition of the test animal organs from each treatment group can be seen. From the picture below, it can be seen that the condition of the organs in the test animal's body no longer experiences swelling or changes in color as in the results of surgery carried out on the seventh day. This condition shows how the mice's bodies can survive after being given a dose of a substance that, under normal circumstances, has the potential to kill the animal; it is called resistance. Previously, test animals were only given a single dose of tar preparations, which caused toxic effects that resulted in changes in behavior and changes in the condition of their digestive organs. The results of the acute toxicity test or LD_{50} show that this dose is relatively harmless; therefore, the test animal's immune system is still able to receive this dose and restore it to its original condition.

The tar generated from gasification at PT SCMPP is not classified as dangerous, but it does have a high iron heavy metal concentration that surpasses the required quality criteria. Each gasifier unit at PT. SCMPP is expected to produce 0.5 tons of tar per hour [7]. Tar contains aromatic hydrocarbons, which can be harmful to other organisms if wasted. Therefore, in order to use tar effectively, more tar waste management is required. Tar quality can be increased with some techniques, including hydro-processing, water cracking, decomposition, steam reforming (using CO2), catalytic cracking, and thermal cracking.



Remarks:
1. Liver
2. Kidney
3. Small intestine
4. Large intestine
5. Bladder

Figure 1. Mice organs on 7th day surgery



Remarks: (1) liver, (2) kidney, (3) small intestine, (4) large intestine, (5) bladder, and (6) stomach

Figure 2. Mice organs on 14th day surgery

4. CONCLUSION

Based on the result and analysis, some heavy metals contained in gasified tar at PT. SCMPP is still within safe limits. Iron (Fe) is the only heavy metal whose content exceeds the quality standards in Minister of

Environment and Forestry No. 6 of 2021. The chemical composition of the trash utilized as a raw material for gasification determine the number of heavy metals. The gasified tar waste at PT. SCMPP does not kill test animals in its acute toxicity test, but it does cause toxic symptoms including lower activity levels and damage to organs. Thus, the LD_{50} value is declared false and uses the highest dose for the determination of toxicity levels, which is 0.16 mL/g BB or equivalent to 160,000 mg/kg body weight. The dose in the Loomis toxicity criteria classified as "relatively harmless" or "non-toxic".

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