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Industrial waste disposal alternatives in the process of aromatic compounds production in petrochemical industry (case study: Nouri petrochemical complex, Asaluyeh, Iran)

Heidari, L., Jalili Ghazizade, M.*, Salemi, A.

Department of Environmental Technologies, Environmental Sciences Research Institute, Shahid Beheshti University, Tehran, P.O.Box. 1983963113, Iran

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ABSTRACT: Application of aromatic compounds has dramatically increased as raw materials in various industries and different factories have been established to produce aromatic compounds. The current research aims at characterizing industrial waste generation in aromatics production process in petrochemical industries and determining the best feasible alternative for waste disposal. For this purpose, the world's biggest aromatic producer, i.e. Nouri Petrochemical Complex (NPC), located in Asaluyeh, Iran, has been selected as case study. Firstly, different waste streams, generated during aromatics production, have been determined through a specific checklist. Spent industrial soil, spent catalyst, spent molecular sieve, and spent Normal-Formyl-Morpholine (NFM) solvent are the most important identified wastes in NPC, with the former being the most generated waste in NPC with a rate of 600 tons per year. Afterwards, the mentioned waste has been sampled and important physicochemical specification such as heavy metals and organic compounds has been measured. Ni, Cu, and As are remarkable trace heavy metals, observed in all kinds of generated waste. In the next step, industrial waste classification and coding has been done, based on different guidelines. Finally different feasible alternatives like material recovery, sanitary landfill, and incineration have been compared, based on conventional economic, technical, and environmental indices. The best feasible waste disposal methods are the extraction of heavy metals from spent catalysts, recycling of spent molecular sieves and spent industrial soil as additives to building materials, and recovery of thermal energy by incineration of spent NFM solvent.

Keywords: aromatic products, spent industrial soil, spent molecular sieve, spent catalyst.

INTRODUCTION

In recent years different industries have been developed to improve the quality of human's life, in turn contributing to severe environmental pollution. Iran possesses the world's second biggest natural gas reserves (Zarinabadi & Samimi, 2012) and the treated gas can be used as a raw material in petrochemical industries. As a result, availability of huge gas resources in Iran has led to considerable development of petrochemical industry. Emission of different gas compounds to the atmosphere is one of the most common environmental aspects of petrochemical industries. As an example, a petrochemical plant chimney releases huge amounts of greenhouse gasses to the air (Lee, 2013). Also, petrochemical industries emit various kinds of heavy metals and hydrocarbons into water sources through untreated wastewater discharge (Wagialla, 2007). Previous researches have

* Corresponding Author, EMail: ma_jalili@sbu.ac.ir

suggested some methods to treat petrochemical plant's wastewater (Marashi & Kariminia, 2012). Furthermore, hazardous waste is another environmental aspect of petrochemical complexes.

Petrochemical industries are usually divided into three groups, namely upstream, midstream, and downstream classes. According to this classification, upstream petrochemical industries generate the most amounts of hazardous wastes (Charmondusit & Keartpakpraek, 2011). Since landfilling of petrochemical hazardous waste may result in contamination of groundwater with hydrocarbons and heavy metals (Musin et al., 2015), different methods have been studied to promote recycling processes in the waste management systems in petrochemical industries (Namasivayam & Senthilkumar, 1998; Wei & Huang, 2001).

Since South Pars is the largest gas field of Iran, comprising 27% of the country's total proved natural gas reserves, most of its petrochemical complexes are located here, at Pars Special Economic Energy Zone (PSEEZ). According to previous studies, petrochemical industries located in PSEEZ are responsible for generation of 11,000 tons of industrial waste in 2004, which will soar beyond 180,000 tons in 2023 (Mokhtarani et al., 2006). According to the Waste Management Act of Iran, generated waste in petrochemical complexes is categorized as industrial waste. This law forces industrial waste producers to decrease their amount of generated waste through optimizing the production process and applying waste recovery methods (DOE, 2005). Furthermore, petrochemical industries should establish environmental management system to obtain ISO1400 standard, where waste management is one of the important parts of this evaluation system (Hogland & Stenis, 2000).

Depending on the products, themselves, along with the production process, large amounts of wastes are being generated in petrochemical industries. Industrial wastes, generated during the production process of olefin, have been studied by Usapein & Chavalparit (2014), who examined the feasibility of reusing, recycling, and reducing methods in order to achieve the best waste management alternative. In another study different industrial waste, generated in the process of Polyvinyl Chloride (PVC) production, was determined. Here, spent caustic flake got considered as waste indicator and different disposal methods such as crystallization, chromatography, incineration, and sanitary landfill were evaluated with regard to their technical, economic, and environmental aspects. Finally, crystallization method was suggested as the best alternative for spent caustic flake disposal (Heidari & Jalili Ghazizade, 2015). A third research investigated three technologies, viz. incineration, gasification, and biogas generation for energy recovery from industrial solid waste by considering technical, economic, and environmental aspects (Murphy & McKeogh, 2004).

Aromatic compounds are one of the petrochemical products with an important role in meeting modern human’s needs, which in turn boosts the consumption of aromatic materials dramatically. Although generated wastewater during the process of aromatic materials production has been characterized by Zandi & Hezarkhani (2007), to the best of the author’s knowledge there has been no research on industrial waste generated in this process so far. It is notable that among 53 active petrochemical complexes in Iran, there are only four units which produce aromatic compounds. The same trend is true elsewhere in the world with only a small proportion of petrochemical complexes dedicated to produce aromatic compounds. As a consequence, the lack of researches on environmental aspects (including industrial wastes) is expected in the literature. Nouri Petrochemical Complex (NPC), the world's largest aromatic material producer, is located on the northern coast of Persian Gulf. Due to other industrial activities in this coast, its ecosystem is contaminated with heavy metals like Ni, Co, Cu, Pb, and Cd, which get accumulated in the sediments and aquatic organisms of Persian Gulf, subsequently affecting the health of people, connected to this body of water for nourishment, work, or recreation (Monikh et al., 2013; Naser, 2013). Therefore with regard to the Integrated Coastal Management Plan (Pak & Farajzadeh, 2007), conceptual design of industrial waste management is essential for different industries in this region such as NPC. As a result the aim of this research is quantitative and qualitative identification of industrial wastes, generated during the production of aromatic materials as well as determination of the best alternative for waste disposal with the lowest environmental risks and economic cost.

MATERIAL AND METHODS

NPC is located in PSEEZ at Bushehr Province, Iran, having a production capacity of 4.5 million ton per year. The main products of NPC are P-xylene, benzene, and ortho-xylene, and heavy hydrocarbons, light hydrocarbons, raffinate, pentane, and liquid gas are by-products of this complex. Figure 1 shows the process of aromatic materials production in NPC.

As the first step of waste characterization, a proper checklist was prepared to identify the waste stream, generated in the process of aromatic materials production. The checklist considered the most important data on waste stream including: name, amount, specification of production unit, frequency of generation, base compounds, and

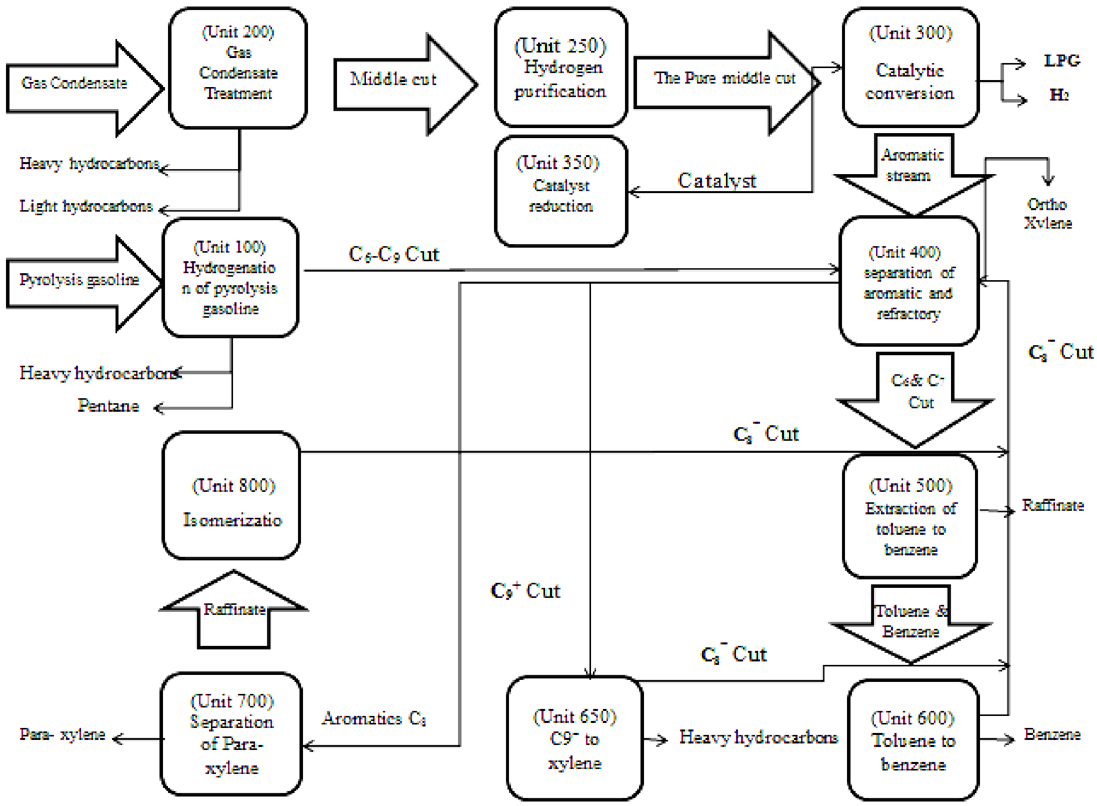

Fig. 1. The process of aromatic materials production in NPC

ongoing management plan. It was filled via visiting the plant and holding interviews with responsible experts of NPC as well as using literature review. Filling the checklist showed that most of the industrial waste in NPC had been generated during overhauls only to be kept in the factory's warehouse without any plan to recycle or dispose it. Among all types of generated wastes in NPC, four types of wastes were remarkable from both perspectives of quantity and hazard potential. They included spent molecular sieve, spent industrial soil, spent catalyst, and spent Normal-Formyl--Morpholine (NFM).

In the next step, sampling of mentioned wastes was planned in accordance with standard methods. Since spent molecular sieve and spent industrial soil are usually stored in factory's warehouse as pile shape, stratified random sampling method was used, wherein the pile was divided into three parts with one sample taken from each section. The spent catalyst was another waste that was being kept in barrels. Since each barrel was filled weekly, systematic sampling over time was selected as the sampling method and three samples of three barrels were taken during a 21-day period. The spent NFM, inherently liquid, was drained from the NFM recovery tank at a rate of 1.5 ton biannually and poured into 220-liter barrels. Therefore, there was no significant difference among the various barrels' contents with the three samples being taken from three different barrels according to simple random sampling method. Afterwards, a main sample was prepared by combining three subsamples for each waste. Ultimately the final four samples were sent to the laboratory for analysis (ASTM, 2012).

In the laboratory, waste density was measured according to ASTM B-962 standard test methods for density of compacted or sintered powder metallurgy (PM) products, using Archimedes' principle (ASTM, 2015). Since heavy metals are common indicators of toxicity in industrial wastes, the most usual trace metals such as As, Ba, Cd, Co, Cr, Cu, Hg, Mn, Ni, Pb, and Zn were considered to be characterized. In the preparation stage, heavy metals in the waste were digested in accordance with EPA method 3050B to get analysis of interest in the solution (EPA, 1996a). Then total concentration of the mentioned elements was measured based on EPA method 6010B, using an Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) technique (EPA, 1996b). Based on automated soxhlet extraction method, the acetone/hexane solvent was passed through solid samples and organic materials were extracted to measure organic pollutants (EPA, 1994). Test of organic compounds were performed according to EPA method 8270E, using both Gas Chromatography/Mass Spectrometry (GC/MS) technique (EPA, 2014) and EPA method 8015D, not to mention nonhalogenated organics using GC/FID (EPA, 2003). The organic compounds were measured with GC Agilent 8970A and a DV5 Column.

Waste classification and coding was conducted using different international guidelines such as Basel Convention, EPA, and UNEP. Finally, different common disposal alternatives (i.e., recycling, landfilling, and incineration methods) were compared together for each waste with regard to technical, economic, and environmental aspects. For evaluation of the mentioned criteria, several approaches can be used in order to comprise different methods. Since various value judgment leads to different results, these criteria were evaluated at four levels (i.e., low, medium, high, and very high) so as to reduce any uncertainty.

RESULTS AND DISCUSSION

The present study identified the most important waste generated in NPC with remarkable industrial wastes being dominantly generated through below processes:

- Exhausted industrial soil used to absorb linear hydrocarbons, called spent industrial soil.
- Residual catalysts in different units crushed physically, called spent catalyst.
- Remaining molecular sieve used to separate P-xylene from other xylenes, called spent molecular sieve.
- Substance obtained from refined of NFM solvent, called spent NFM solvent. The NFM solvent was applied to extract benzene and toluene from other hydrocarbons.

Table 1 shows the physical characteristics and basic compounds of four types of waste and responsible process units. As seen in Table 1, spent molecular sieve and spent NFM solvent were generated only in one process unit; however, several units were responsible to generate spent industrial soil and spent catalyst. According to this table, the highest amount of generated waste in NPC was spent industrial soil with a rate of 600 ton per year. Frequency of waste generation in NPC varied for different wastes from one week to one year. Measured volumetric mass of the wastes showed that spent catalyst had the highest density thanks to its metallic structure, whereas the spent NFM solvent with polymeric structure turned out to be the lightest one. Attention to physical features of wastes shows that all kinds of four identified wastes had a solid form. It should also be mentioned that spent NFM solvent is inherently semi-solid at the beginning of production yet after being exposed to air it becomes a tar stiff shape.

Table 2 shows total concentration of heavy metals in different wastes, compared to Total Threshold Limit Concentration (TTLC) first introduced by California Code of Regulations (CCR). TTLC was applied as primary indicator of waste toxicity characteristic in order to know whether more detailed analysis (e.g. Total Characteristic Leaching Procedure (TCLP) or Soluble Threshold Limit Concentration (STLC)) is needed or not. Results indicated that Ni concentration in spent catalyst exceeded the TTLC regulatory limit of 2000 mg/kg, absolutely considered as hazardous waste. Also Ni in spent industrial soil and As in spent NFM solvent can be responsible to put these wastes into hazardous waste category, if the entire concentration of mentioned constituent is soluble in Waste Extraction Test (WET).

Table 1. Physical characteristics of processed wastes in Nouri Petrochemical Complex

Industrial waste	Production unit number	Amount (tonne/year)	Frequency	Basic compounds	Density (g/L)	Physical features
Spent industrial soil	400, 600, 800	600	Annual	Silica and alumina	829	Black powder
Spent catalyst	100, 300, 800	11	Weekly	Platinum and alumina	1485	Cylindrical particles, cream, grayish
Spent molecular sieve	700	133	Decennial	Silica and alumina	756	Cylindrical particles, white
Spent NFM solvent	500	3	Half-yearly	Polymer	502	Black sticky substance

Table 2. Total concentration of heavy metals (mg/kg) in processed wastes in Nouri Petrochemical Complex

Element	Spent industrial soil	Spent Catalyst	Spent molecular sieve	Spent NFM solvent	TTLC[1] regulatory level (CCR, 2016)
As	<1	<1	<1	5	50
Ba	98	5	5	2	10000
Cd	<0.1	<0.1	<0.1	3	100
Co	4	61	<1	<1	8000
Cr	6	4	11	15	500
Cu	21	132	3	7	2500
Hg	<0.1	<0.1	<0.1	<0.1	20
Mn	381	4	14	646	-
Ni	429	257600	2	7	2000
Pb	6	<1	<1	6	1000
Zn	29	245	12	60	5000

1. Total Threshold Limit Concentration

Table 3. Qualitative analysis of organic compounds in processed wastes in Nouri Petrochemical Complex

Compounds	Spent industrial soil	Spent catalyst	Spent molecular sieve	Spent NFM solvent
Benzene, 1-methy1-4-(phenylmethyl)	N.D	N.D	N.D	N.D
Benzene, 1-methyl-2-[(3-methylphenyl)methyl]	N.D	N.D	N.D	N.D
Benzene, 1,1'-methylenebis[4-methyl]	N.D	N.D	N.D	N.D
Naphthalene, 1,2,3-trimethyl-4-propenyl	N.D	N.D	N.D	N.D
Methanone, bis-(3-methylphenyl)	N.D	N.D	N.D	N.D
Ethanol, 2,2'-bis-(methylimino)	N.D	N.D	N.D	N.D
Chloroiodomethane	N.D	D	D	N.D
N-Formylmorpholine	N.D	N.D	N.D	D
Methane, diiodo	N.D	D	D	N.D
Styrene	N.D	N.D	N.D	N.D
ethyl benzene	N.D	N.D	N.D	N.D
Benzene	N.D	N.D	N.D	N.D
Naphthalene	N.D	N.D	D	N.D
Acenaphtylene	N.D	N.D	N.D	N.D
Acenaphthene	N.D	N.D	D	N.D
Fluoranthene	N.D	N.D	D	N.D
Pyrene	N.D	N.D	D	N.D
Toluene	N.D	N.D	N.D	N.D
Benzene, 1,1'-ethylidenebis	N.D	N.D	N.D	N.D
Phenanthrene	N.D	N.D	N.D	N.D
Trimethylbenzene	N.D	N.D	N.D	N.D
2-Phenylnaphthalene	N.D	N.D	N.D	N.D
Decane	N.D	N.D	D	N.D
Tetradecane	N.D	N.D	D	N.D
Hexadecane	N.D	N.D	D	N.D
Benzene, 1-methyl-3-(2-phenylethenyl)	N.D	N.D	D	N.D

ND: Not Detected; D: Detected

Table 3 presents the qualitative analysis of organic compounds. Among 26 examined organic compounds, only 11 compounds were detected in all samples. As it can be seen, there was no detectable organic compound in spent industrial soil. To justify this phenomenon, it should be said that nitrogen gas and water vapor had been passed through the industrial soil before being discharged as some sort of waste and that this process led to hydrocarbons removal, in turn rendering spent industrial soil free of hydrocarbons compounds. Only N-Formylmorpholine could be detected in spent NFM solvent and the chloroiodomethane and methane (diiodo) were detectable in the spent catalyst. However, a range of polycyclic aromatic and alkanes compounds were detected in spent molecular sieve. It should be noted that the use of molecular sieve was to separate P-xylene from other xylenes. For this purpose a mixture of xylene types got passed through the surface of molecular sieve to absorb P-xylene. Absorbed P-xylene in molecular sieve can be separated by using Para Diethyl Benzene solvent. Since the different organic compounds were in contact with the molecular sieve, various organic compounds remained in spent molecular sieve. Thus in the next step, the quantitative analysis of organic compounds was carried out for spent molecular sieve, the result of which is presented in Table 4. It can be seen that naphthalene had the maximum amount of organic compounds in spent molecular sieve.

In the next step, all identified wastes got classified and coded according to different standards. Table 5 shows the classification and coding of the wastes according to standards of the Basel Convention, EPA, and UNEP.

Table 4. Results of quantitative analysis of organic compounds in spent molecular sieves

Compounds	Benzene	Toluene	Ethyl benzene	Xylens	Styrene	Naphthalene	Acenaphthylene	Acenaphthene	Flourene	Phenanthrene	Anthraene
Concentration (μg/g)	<0.1	<0.1	<0.1	<0.1	<0.1	1.1	<0.2	0.9	<0.2	N.D	N.D
Compounds	Flouranthene	Pyrene	Benzo(a)anthraene	Chrysene	Chrysene	Benzo (k)flouranthene	Benzo (a)Pyrene	Indeno(1,2,,3-cd)pyrene	Dibenzo(a,h)anthraene	Benzo(g,h,i)perylene	_
Concentration (μg/g)	<0.2	<0.2	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D	_

N.D: Not Detected

Table 5. Classification and coding of processed wastes in Nouri Petrochemical Complex

Waste type	Basel Convention			EPA			UNEP	
	Attach 1 (Y)	Attach 3 (H)	Attach 8 (A,B)	K	F	Nature T-C-R-I	T.M	T.I
Spent industrial soil		H6.1 H11 H12		K170		T	A	F
Spent catalyst		H11 H12 H6.1 H4.1		K172 K171		I-T	A	F
Spent molecular sieve	Y9	H6.1 H4.1	A2030	K170		T	A	F
Spent NFM solvent	Y42	H4.1	A3140		F003 F004 F005	I-T	C	F

T.M.: Type of Material; T.I.: Type of Industrial soil

Table 6 compares waste disposal methods based on technical, economic, and environmental indices. According to this table, landfilling is a simple method that does not need water or other chemicals. However, necessity of a large area of suitable land is the most important weakness of this method. In addition, it may require complementary processes (i.e., stabilization and solidification) to reduce toxic emission from industrial landfills. This table considers some recovery methods, such as the use of spent industrial soil and spent molecular sieve in construction materials production, the extraction of metals from spent catalyst, and the recycling of spent NFM. With the exception of spent NFM, which consists of different organic compounds, all other wastes are made of mineral composition, thus Waste-to-Energy method (incineration) has been studied only for spent NFM solvent.

Table 6. Comparison of different methods as feasible alternatives for Nouri Petrochemical Complex waste disposal

	Indicators	**Industrial soil**			**Catalyst**			**Molecular sieve**			**NFM solvent**		
		SL	**R**	**I**	**SL**	**R**	**I**	**SL**	**R**	**I**	**SL**	**R**	**I**
Technical	Technological and operational complexity	L	L	Because of the minerals make up the main part of it, this method is not suitable.	L	H	Because of the minerals make up the main part of it, this method is not suitable.	L	L	Because of the minerals make up the main part of it, this method is not suitable.	M	According to present study is not possible	H
	Efficiency	M	M		M	H		M	H		M		H
	Need to land	VH	L		VH	L		VH	L		VH		L
	Need to water	L	L		L	M		L	L		L		Does not have
	Need to consumables / chemical	M [A]	L		M [A]	H		M [A]	Does not have		M [A]		M [A]
Economic	Investment and operating costs	L	L		L	H		L	L		L		VH
	The value of recycled products	L	M		L	VH		L	M		Does not have		M
environmental	The potential release of dangerous pollutants	M	L		M	L		M	L		M		M
	Material and energy recycling	L	H		L	H		L	H		L		H

[A] Stabilization and solidification process is necessary for disposing these wastes
VH: Very high; **H**: High; **M**: Medium; **L**: Low.
R: Recycling; **SL**: Sanitary Landfill; **I**: Incineration.

The best method of waste disposal was selected in accordance with waste management comprehensive hierarchy, wherein the first priority is resource recovery (both material and energy) and the last one is land disposal; therefore, the recycle method was investigated firstly, followed by the possibility of recovery energy. Eventually, landfilling option was examined as the best management method for each waste.

As shown in Table 6, recovery of the spent industrial soil was selected as the best feasible method in this study. In other similar studies, spent industrial soil was used in a building material, e.g. brick and ceramic. This method can improve the quality of produced brick and ceramic and is capable of controlling environmental impacts of spent industrial soil disposal (Mymrine et al., 2013; Eliche-Quesada, 2015; Eliche-Quesada & Corpas-Iglesias, 2014). As a matter of fact, the clay, used in bricks or ceramics, can stabilize heavy metals of the spent industrial soil and prevent any emission into the environment.

In this study, the best waste management method turned out to be recovery of the spent catalyst, in which some metals must be extracted from spent catalyst. Nevertheless, this method is very expensive, even though the high value of recycled metals can compensate for some process expenditures. On the other hand, extraction of metals partially controls the emission of heavy metals into the environment and can decrease the volume of remaining waste, as well. Different methods were investigated for the extraction of metals from wastes, including hydrometallurgy, pyrometallurg, and metal extraction by means of acid and microwave radiation (Asghari, 2013). Furthermore, biodegradation methods of heavy metal extraction has been recently applied to increase the efficiency of metal extraction (Shahrabi-Farahani et al., 2014; Jafarifar et al., 2005; Mishra & Rhee, 2010).

Recovery of the spent molecular sieve was selected as the best waste management method. It can be reused instead of cement or sand to promote mortar specifications, regarding zeolitic structure of spent molecular sieve (Su et al., 2000; Al-Jabri et al., 2013). In addition, available carbon inside the spent molecular sieve could be utilized to upgrade the quality of products (Zhang et al., 2014). This method also decreases toxic emissions due to pollutant stabilization via other building materials in mortar (e.g. cement).

There was no method for recycling spent NFM in the best of the author's knowledge, yet thanks to hydrocarbon structure of spent NFM solvent, it can be used as a thermal energy source. Hence, the use of an incinerator to burn the spent NFM solvent is recommended as the best waste disposal method. The incinerator must be equipped with a control system to prevent pollutants' emission into the atmosphere. It was proven that the volume of leftover ash in the incinerator had been much less than the initial waste.

CONCLUSION

Since the most important step in industrial waste management process is waste characterization, the present research focused on identification of generated waste stream in NPC, in both quantitative and qualitative terms. Results showed that about 750 tons of industrial waste was annually generated in NPC and currently stored in the factory's warehouse. The most important wastes generated in the process of aromatic substances production included spent industrial soil, spent catalysts, spent molecular sieves, and spent NFM solvent. What is more, Ni, Cu, and As were the most dominant trace metals in the sampled wastes. Laboratory analyses showed that spent catalyst had to be considered as hazardous waste; however, more detailed risk assessment study is still necessary for spent industrial soil and spent NFM. Measuring organic compounds indicated that there were no organic pollutants in spent industrial soil and spent catalyst. These kinds of contaminants were only detected in spent NFM solvent due to the composition of its raw material and in spent molecular sieves because of its functional role. Waste classification showed that all generated wastes in NPC were categorized as hazardous waste, according to different standards such as Basel Convention, EPA, and UNEP; therefore, special checks and inspections should be considered during all stages of waste management from generation to final disposal. The 3R strategy was used to propose the best method of waste disposal by considering the priorities in a hierarchy of comprehensive waste management methods (viz., material recycling material, energy recovery, and landfilling). Results showed that the use of spent industrial soil and spent molecular sieves in building materials construction, recovery of valuable metals from spent catalyst, and thermal recovery from NFM solvent can be considered as the most proper disposal methods in NPC industrial wastes management. However, more precious researches should be conducted in laboratory and pilot scale in order to implement the proposed methods. In this regard, the optimum percentage of spent industrial soil and spent molecular sieves must be determined as an additive in building materials. What is more, engineering characteristic of the constructed building materials (i.e., brick, concrete, etc.) must be examined. In the next stage, environmental impacts of this method should be monitored. In addition modern methods for extraction of heavy metals can be surveyed to increase the recycling rate of spent catalyst. The amount of toxic emissions from the combustion of spent NFM is another point that should be further investigated as well as the ash nature and the optimal method of disposal. Further examination is recommended to asses other harmful aspects of wastes, such as toxicity, flammability, and corrosiveness.

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