Hindawi Publishing Corporation
Journal of Waste Management
Volume 2013, Article ID 278618, 7 pages
http://dx.doi.org/10.1155/2013/278618

Research Article

Development of a Technology for Treating Wastewater Contaminated with Nitric Acid

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Received 19 March 2013; Accepted 3 June 2013

Academic Editor: Nigel J. Horan

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The production process of nitroaromatic hazardous compounds, with the generation of acidic wastewater, represents a significant danger for the health and safety of the workers and the environment. The present study is focused on the development of an efficient installation to treat acidic wastewater resulting from the synthesis process of nitroaromatic compound, considering workers safety and environmental criteria. In this research, a detailed study of the different alternatives that can be used for effective and safe treatment of acidic wastewater was performed. The analysis of several technological schemes for the acidic wastewaters neutralization and the selection of the most feasible alternative from a technical-economic point of view were carried out. The simulation and mathematical modeling developed in this research represent a significant advance in the knowledge of this process for working in a much more secure form. The technological scheme of the process was defined, and the design of the main and auxiliary equipment as well as the piping system was carried out using different computational programs. Finally, this paper proposes a technological design for the treatment of acidic wastewater generated by the production process of nitroaromatic compound, which represents the basic criteria for the further design, construction, and equipment installation of the plant.

1. Introduction

In Cuba, the environmental problems represent one of the major concerns since 1981 when the law of environmental protection and rational use of natural resources was approved. Several studies to improve the treatment of hazardous wastes generated from explosive production industry have been conducted.

The production process of nitroaromatic hazardous compounds, with the generation of acidic wastewater, represents a significant danger for the health and safety of the workers and the environment. This wastewater containing solid particles of the nitroaromatic compounds and nitric acid in a concentration range between 58 and 62% constitutes a potential risk to the environment.

The present study is focused on to develop an efficient installation to treat acidic wastewater resulting from the synthesis process of nitroaromatic compound, considering workers safety and environmental criteria.

2. Materials and Methods

In this research, a detailed study of the different alternatives that can be used for effective and safe treatment of acidic wastewater was performed. The analysis of several technological schemes for the acidic wastewaters neutralization and the selection of the most feasible alternative from a technical-economical point of view were carried out.

2.1. Technoeconomic Analysis of Alternatives. A possible alternative is based on nitric acid (HNO_3) recovery, where the acid waters are concentrated on achieving a nitric acid that could be reused in the manufacturing process. The use of this alternative is recommended for a process having significant amount of acids. A considerable reduction of the production cost (by raw material concept) can be obtained with this alternative. This alternative has many advantages; however, a major economic disadvantage could be highlighted in that the acquisition of a recovery plant is required.

Other alternatives for the treatment of this type of effluent are based on the neutralization by the use of different products. It has been reported that the best neutralization compounds are ammonia, urea, carbonates, or oxides of alkali metals and alkaline earth [1, 2].

2.1.1. Carbonate Rocks and Metal Oxides. For this method, calcium carbonate, calcium oxide, and dolomite can be used. In the central region of Cuba, large natural reserves of these compounds can be found, making the production process less expensive. From a comparison of these compounds, the dolomite represents the most suitable compound due to its least cost. Besides, the technology does not present major technological complications. Nevertheless, due to the great demand that the dolomite has in the glass production, an insufficient amount of this compound for the use in the neutralization process could be observed. With respect to the product obtained, the calcium carbonate and calcium oxide as well as the dolomite may have some application in agriculture. However, due to Cuba climate conditions, the use of calcium carbonate, calcium oxide (in solution or granulated form), and dolomite is not recommended. Moreover, in low calcium soils (not the Cuban situation) the calcium nitrate is used. On the other hand, the potassium carbonate which is also a compound very often applied must be imported.

2.1.2. Urea and Ammonia. The use of ammonia and urea shows higher production cost in comparison with carbonate rocks, but analyzing the possible use of the final product, this alternative has the best results. Specifically, the neutralization with ammonia obtaining ammonium nitrate shows the greatest advantages because this fertilizer has a significant demand in our country, incorporating 35% of nitrogen to the Cuban soils.

The neutralization with ammonia represents the best alternative because it offers a viable solution for the acidic wastewater treatment obtaining a final product with demand in the agriculture. Although obtaining ammonium nitrate pearl has several advantages, the equipment and production costs are very high. The option of obtaining the liquid fertilizer 32-0-0 represents the best alternative because a feasible economic balance was found. The liquid fertilizer 32-0-0 (called by its composition) is a high nitrogen content fertilizer with three split nitrogen sources and 32% of total nitrogen. Moreover, it can be dosed to the ground together with irrigation water (fertirrigation), it is noncorrosive, and it is not possible to solidify at decreasing temperatures. Furthermore, a significant advantage for the plant development can be observed preventing damage to the rootlets by the intensive use of dry fertilizers. The disadvantages are outweighed by the overall efficiency and are also relatively a simple solution. This technique can be applied in a wide range of situations with regard to crop types as well as the soil and water characteristics according to [3–5].

2.2. Definition of the Technological Scheme. Considering that the wastewaters (raw material) which come from the synthesis plant with a mass flow of 1500 kg/d contain solid particles of 80 μm of average diameter and a concentration of nitric acid between 58 and 62%, a specific technological process is proposed. The flow diagram of the wastewater treatment plant is shown in Figure 1.

The technological scheme defined consists of three basic steps: cleaning or preparation of residual, neutralization step, and obtaining the final product. The same is described: during the production of aromatic nitro compound, the acidic wastewater contains small amounts of solid particles, which if passed to the neutralization stage, it would jeopardize the process, resulting in the occurrence of violent reaction with high heat release. A sedimentation-filtration system was used for purifying the acidic wastewater. In the settling section to precipitate the dissolved solids in the wastewater, a long time of settling was required. As the process will work at batch conditions, a suitable volume settler with the greatest possible area of sedimentation was designed. This equipment consists of two rectangular chambers where the first one (largest) was used as a sedimentation region, in which the acidic residual waters that come from the synthesis plant of nitroaromatic compound arrives for a pipeline that discharges in its depth. After seven days, the first chamber is filled and the liquid passes smoothly per cloth filter through the second chamber. Subsequently, when the second chamber has a residual volume stored for one day, the pump installed in the surface of the dispenser extracts and pushes it into the filter. During the sedimentation, the 75% of the solids are eliminated, and the remaining particles are trapped in the thin fabric nutsch filter. The settler sludge is unloaded by hand each certain time by the top of the settler.

In the filtration step, a nutsch filter was employed as it is recommended for the effective separation of much diluted suspensions. It operates at discontinuous and vacuum conditions. It has the advantage of facilitating a complete and uniform washing of the sediment, recovers the solids, and provides safety for explosives handling. More details regarding the mechanical characteristics of this type of filter can be found in Rosabal and Valle [7].

When the filtrate is obtained, it is passed until the neutralization stage flows the obtained liquid by gravity to a batch reactor with gas bubbling operating at atmosphere pressure. This equipment consists of a stainless steel cylinder 304, uncoated, in which the acid solution will be discharged and the neutralizing NH_3 (g) will be provided by the background through a bubbler of stainless steel coated with Teflon.

In the neutralizer, the next chemical reaction takes place: $NH_3 + HNO_3 \rightarrow NH_4NO_3$. The violent conditions that occur when these two substances react are placated with the addition of an ammoniacal solution containing 15% of NH_3 (g) in which the medium is passive, decreasing the reaction of ammonia gas and the acid. As a result of this reaction and the heat release that takes place, the obtained water vapors contain small amounts of ammonia and formed solution.

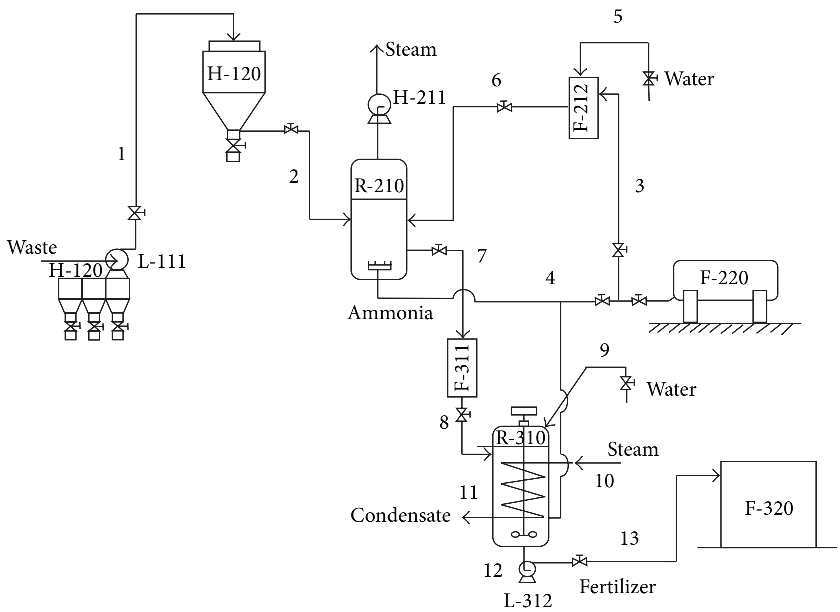

Figure 1: Technological scheme of the proposed wastewater treatment plant. H-110: settler; H-120: nutsch filter; R-210: bubbling reactor; F-220: ammonia bullet; R-310: stirred tank of fertilizer preparation; F-320: fertilizer storage tank; L-111: residual pump (HNO_3); H-211: extractor; F-212: ammonia solution tank; F-311: intermediate storage tank of ammonia nitrate; L-312: fertilizer pump.

These slightly contaminated vapors (1.5–2% NH_3 and <1% HNO_3) are discharged to a suitable height using an extractor, avoiding the negative impact on the environment and human health.

The ammonium nitrate obtained has a concentration of 80% and flows by gravity to an intermediate storage tank where one part of the solution is stored and the other part continues flowing until an agitation tank that include a heating coil. In the stirred tank, liquid fertilizer known for its land application as fertirrigation and for its composition and 32-0-0 is prepared. A propeller type agitator was selected, and considering the process requirements, this equipment needs to be manufactured in stainless steel. Equally, the cylindrical container with conical bottom and the coil used in the process must be constructed of stainless steel.

In order to obtain the fertilizer mentioned previously (32% of total nitrogen), the ammonium nitrate (51%) is mixed with the required amounts of urea (30%), phosphoric acid (0.2%), ammonia (0.5%), and water (19%). To carry out this research, the archived tables obtained from the nitrogenized fertilizer plant of Cienfuegos (a Cuban province) were consulted. The fertilizer formed has excellent characteristics when applied to cultivated soil. Other significant advantage can be shown from its use due to his non-corrosive characteristics, being used in pipelines, irrigation equipment and storage of carbon steel or aluminum. Furthermore, this type of fertilizer can be dosed to the ground according to the crops need and does not represent danger by the crystallization effect.

To achieve a better effectiveness in the treatment of contaminated wastewater, several experiments at laboratory scale were conducted to determine the most important reaction parameters such as reaction temperature, amount of evaporated water, and reaction time. The installed setup at laboratory scale involves the following equipment: ball of two-necked flask (500 mL) with boilers, water bath, gases trap, three-necked flask (500 mL), thermometer (scale 0–300°C), and gases bubbler (Dean Stark) [8].

2.3. Process Simulation. Considering the results reported by Hidalgo et al. and the use of the appropriate software which allowed modeling, simulating, and scaling of the chemical reaction stages, mathematical methods and procedures were developed for evaluating and predicting the behavior of dangerous reactions, achieving integrated designs of the reactors that minimize the consumption of material resources, reducing waste disposal to the environment, and ensuring more security when working with hazardous substances [9].

Simulation was accomplished with a process dynamic model. The model is constituted by the component's balance, the energy balance, the process kinetics, and the electric load balance. The kinetics of the process was limited by the gas-liquid mass transfer. To determine the mass transfer coefficient, the equations reported by [10–14] were used in the present research (Table 1).

The main equations considered to describe the model can be expressed as follows.

Component mass balance

$$\frac{dN_i}{dt} = \sum R_j V_l, \quad (1)$$

where N_i is the number of moles of i components (mol) and $i = NH_3, HNO_3, NH_4OH, H_2O, NH_4NO_3$; R_j represents the terms of rate (mass transfer rate, kinetic reaction rate, and evaporation rate) expressed in mol h^{-1}, and V_l is the volume of liquid in l.

Table 1: Rate terms used in the mass balance equations[a].

Components (i)	Evaporation rate	Mass transfer rate	Reaction rate
NH_3	$Kv_i(p_i - py_i)$	$\mathrm{kla}(c_{eq} - c)$	$-kc_{NH_3}c_{HNO_3}$
HNO_3	$Kv_i(p_i - py_i)$	—	$-kc_{NH_3}c_{HNO_3}$
NH_4NO_3	—	—	$kc_{NH_3}c_{HNO_3}$
H_2O	$Kv_i(p_i - py_i)$	—	—

[a]Note: k_{vi} and p_i are the evaporation rate constant (mol h^{-1}) and the equilibrium pressure (atm) of components (i), py_i is the vapor pressure represented by the molar fraction of vapor (atm), kla is the volumetric coefficient of mass transfer (h^{-1}), and k represents the kinetic constant (mol^{-1} L h^{-1}).

Energy balance

$$\frac{dT}{dt} = \frac{1}{mC_p}\sum Q_k, \tag{2a}$$

where T, t, and m are temperature (°C), time (h), and reaction mass (kg), respectively. C_p represents specific heat of liquid (kJ kg^{-1}°C^{-1}), Q is heat rate (kJ h^{-1}), and k symbolizes the input (in), reaction (r), solution (s), and evaporation (v) heat.

Consider

$$\begin{gathered} Q_i = f_{NH_3} C_p g_{NH_3} T_{\mathrm{input}}, \\ Q_r = 104.5 R_{NH_3} V_l, \\ Q_s = 34.6 f_{NH_3}, \\ Q_v = -\left(23.33 Rv_{NH_3} + 40.67 Rv_{H_2O} + 39.09 Rv_{HNO_3}\right), \end{gathered} \tag{2b}$$

where f_{NH_3}, $C_p g_{NH_3}$, and R_{NH_3} are mass flow (kg h^{-1}), specific heat in the gaseous phase (kJ kg^{-1}°C^{-1}), and reaction rate (mol h^{-1}) of NH_3, respectively; T_{input} is the initial temperature (°C), and Rv_{NH_3}, Rv_{H_2O}, and Rv_{HNO_3} are the evaporation rates (mol h^{-1}) of NH_3, H_2O, and HNO_3, respectively.

Charge balance

$$\begin{aligned} c_{HNO_3} + 10^{\mathrm{pH}+14} + \frac{c_{NH_4OH}}{1 + 10^{4.75+\mathrm{pH}-14}} \\ = 10^{-\mathrm{pH}} + \frac{c_{NH_4{}^+ + NH_3}}{1 + 10^{-9.25-\mathrm{pH}}}, \end{aligned} \tag{3}$$

where c_{HNO_3}, c_{NH_4OH}, $c_{NH_4{}^+}$, and c_{NH_3} represent the concentrations of HNO_3, NH_4OH, $NH_4{}^+$, and NH_3, respectively.

3. Results and Discussions

3.1. Design of the Proposed Wastewater Treatment Plant. The neutralization of waste from the synthesis the nitroaromatic compound using ammonia is the most feasible, because it combines a lower economic cost and high demand of the product (ammonium nitrate) in agriculture. The ammonium nitrate obtained will be used as a fertilizer in liquid form. The technological scheme alternative was defined for obtaining the called *fertirrigation* due to its several advantages for the land application.

The technological scheme of the process based on fertirrigation (Figure 1) was defined, and the design of the main and auxiliary equipment as well as the piping system was carried out using different computational programs and for determining the thickness of insulation required in each case. The equipment layout of the proposed plant was carried out. For this purpose, the characteristics of the gravity for the different transport fluids involved in the process were taken into account to place the equipment in each corresponding level of the plant. Moreover, the compaction of the interrelated equipment without affecting the workers comfort and the environment protection as well as establishing the conditions for the chemical safety handling and the process management without risk of accidents was considered [8].

An approximate design of the most important equipment of the fertirrigation process was done taking into account the mass and energy requirements and the design equations of the equipment involved. The overall wastewater treatment plant included a sedimentation tank, nutsch filter, bubbling reactor, ammonia bullet, stirred tank of fertilizer preparation, fertilizer storage tank, residual pump (HNO_3), extractor, ammonia solution tank, intermediate storage tank of ammonia nitrate, and fertilizer pump.

3.2. Reaction Parameters Obtained by Simulation. Figure 2 represents the behavior of the process obtained by simulation. An increment of the mass of ammonium nitrate was observed with the time. On the other hand, a substantial decrease of the mass of nitric acid and water was obtained in correspondence with the evaporation process and the chemical reactions involved. Throughout the whole experiment, an acid pH (1) and a temperature in a narrow range of values (120–130°C) were observed, in line with the self-controlling effect that produces the evaporation.

It was experimentally determined that reaction temperature between nitric acid and ammonia varies in a range between 120 and 130°C, and the material and energy balances were made for a production capacity of 600 ton/y. The simulation results are well adjusted with the obtained laboratory data in regards to the prevalent operating conditions of the process and the obtained conversion.

The results of simulation agree with the results of laboratory in regards to the prevalent operating conditions and the obtained conversion. At the highest reaction temperature (around 130°C), a pH approximately of 1, an 80% of conversion, and little losses of nitric acid and ammonia (less than a 3% of the total) were obtained. Moreover, it was observed that the heat of reaction is enough to concentrate the solution and the process shows good level of controllability, in regards to the temperature behavior.

3.3. Economic Assessment. The economic assessment was addressed to determine static economic indicators such as Total Capital Investment (TCI), total production cost (TPC), and annual gross profit (AGP) as well as dynamic economic indicators such as net present value (NPV), payback period (PP), and internal rate of return (IRR).

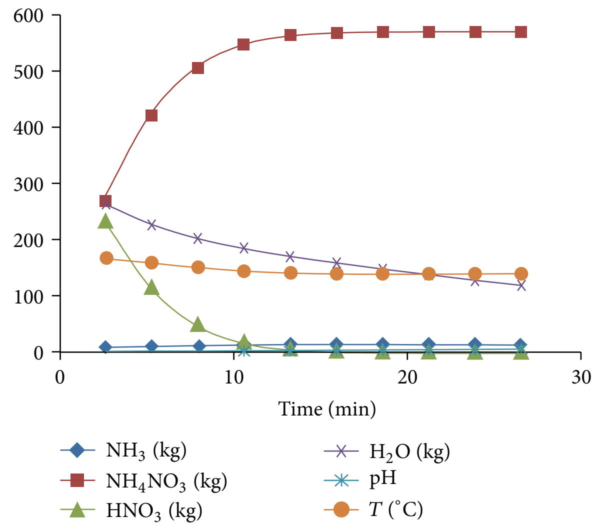

FIGURE 2: Effect of the reaction time and the temperature on the behavior of synthesis compounds.

TABLE 2: Equipment investment costs of the wastewater treatment plant.

Equipment	Investment costs (US$)	Reference
Product mixed tank	93 079	[6]
Sedimentation tank	124 933	[6]
Reactor	108 385	[6]
Filter	2 316	[6]
Storage tank 1	9 514	[6]
Storage tank 2	29 785	[6]
Storage tank 3	96 251	[6]
Centrifuge pump 1	6 067	[6]
Centrifuge pump 2	7 308	[6]
Fan extractor	4 413	[6]

The updated equipment investment costs (CEIndex-JUNE/2012 = 729.9) of the wastewater treatment plant (Table 2) as well as the estimation of the TCI by percentage of delivered equipment method (Table 3) have been outlined [15]. High values of Total Capital Investment (US$ 1 446 296) are mainly attributed to equipment investment cost (US$ 368 014) where the reactor and sedimentation tank represent 63% of the total equipment investment cost.

An integrated economic analysis of the proposed technology with the synthesis plant of a nitroaromatic compound was carried out. For this integration, the values of capital spending (US$) based on the total cost of raw materials and Annual sales (US$) reported in 2012 by the nitrogenized fertilizer plant are shown in Table 4 (data reported by personnel of the plant).

Considering the proposed wastewater treatment technology, static and dynamic economic indicators were evaluated (Table 5).

TABLE 3: Estimation of the total capital investment of the wastewater treatment plant.

Parameters	Fraction of purchased equipment	Calculated values (US$)
Direct costs		
Purchased equipment	—	368 014
Purchased equipment installation	0.2	73 603
Instrumentation and controls	0.15	55 202
Piping (installed)	0.25	92 004
Electrical systems (installed)	0.08	29 441
Buildings (including services)	0.1	36 801
Yard improvements	0.05	18 401
Service facilities (installed)	0.5	184 007
Total direct costs	—	857 473
Indirect costs		
Engineering and supervision	0.1	36 801
Construction expenses	0.2	73 603
Legal expenses	0.04	14 721
Contractor's fee	0.15	55 202
Contingency	0.22	80 963
Total indirect costs	0.71	261 290
Fixed capital investment (FCI)	—	1 118 763
Working capital (WC)	0.89	327 533
Total capital investment (TCI)		1 446 296

TABLE 4: Annual sales and capital spending based on the raw materials cost of the wastewater treatment plant.

(a)

Products	Annual sales (US$)
Ammonium nitrate	600000
Total annual sales (US$)	600000

(b)

Materials	Price (US$/m^3)	Annual amount (ton/a)	Annual cost of raw materials (US$/a)
Nitric acid	115	78	8970
Urea	159	180	28620
Phosphoric acid	792	6	4752
Capital spending as total annual cost of raw materials			42342

A high annual gross profit was observed for the proposed wastewater treatment plant (US$ 358 675), which demonstrates that a positive economic assessment can be achieve using the current technology. The profiles of the net present value (NPV) obtained for the proposed wastewater treatment plant are shown in Figure 3.

At the same time, for this technology a payback period of 3 y and a return on investment of 22% y were achieved. It is necessary to remark that for these profitability measures,

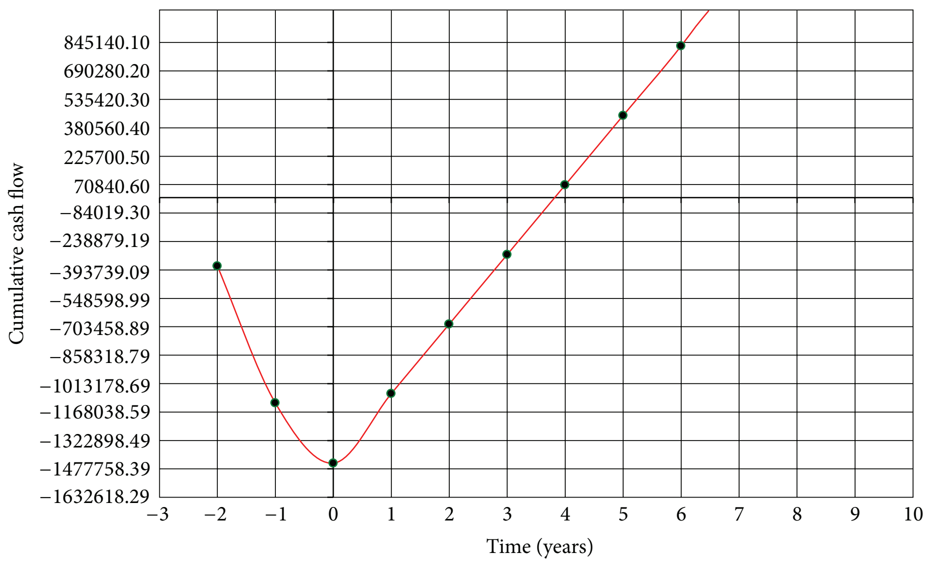

FIGURE 3: Project cash flow diagram of the proposed wastewater treatment plant.

TABLE 5: Economic assessment using static and dynamic economic indicators*.

Economic indicators	Wastewater treatment plant
Annual sales (US$)	600000
Annual total production cost (TPC) (US$/y)	185387
Annual depreciation (US$/y)	55938
Annual gross profit (AGP) (US$)	358675
Return on investment, aver. (ROI) (%/y)	22
Payback period (PP) (y)	3
Net present value (NPV) (US$)	924399
Internal rate of return (IRR) (%)	26

*Considering an annual depreciation factor of 0.10 (10 years of project's health life).

the time values of money (inflation) were not included. An internal rate of return (IRR) of 26% was acquired from the present technology. A residual treatment plant project that reaches an IRR superior of 10% can be considered profitable [16].

4. Conclusions

From this study, was determined that the use of neutralization with ammonia, for obtaining the fertilizer (32-0-0), represents a feasible alternative to treat acidic wastewater generated during the production process of nitroaromatic compound from technical, economic, and environmental point of views.

Simulating and modeling procedures were done due to the lack of available information about the chemical reaction parameters that take place during the formation of ammonium nitrate. The simulation and mathematical modeling developed in this research represent a significant advance in the knowledge of this process for working in a much more secure form. This paper proposes a feasible technological design for the treatment of acidic wastewater generated by the production process of nitroaromatic compound, which represents the basic criteria for the further design, construction, and equipment installation of the plant.

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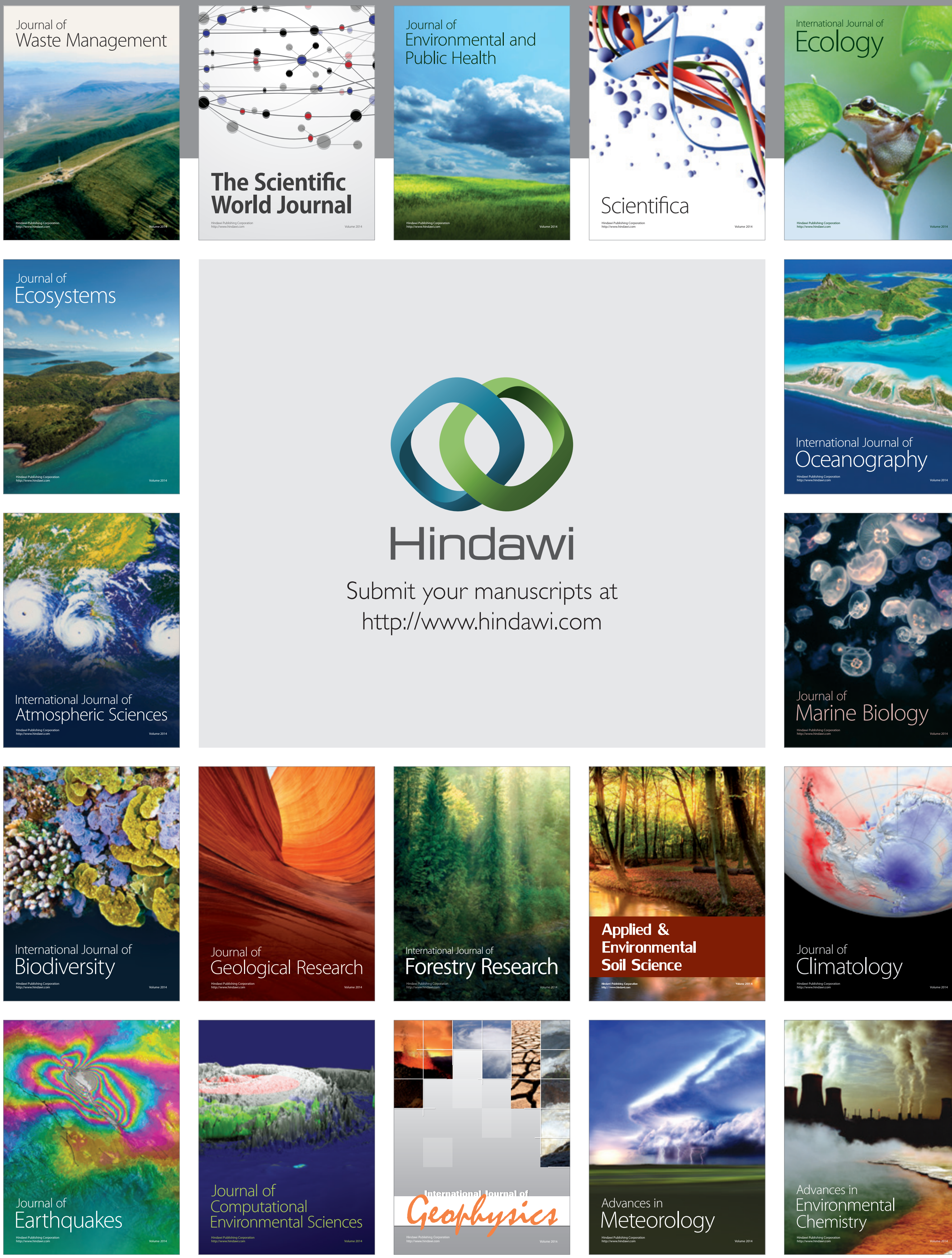

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