

Recuperação de resíduos de etanol e formol gerados em laboratório de zoologia

Recovery wast ethanol and formol reuse generated in zoological laboratories.

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Resumo

Este trabalho apresenta processos de recuperação de resíduos de formaldeído e etanol gerados em laboratórios. O formaldeído com concentração desconhecida e etanol residual de teor 70 °GL foram coletados no laboratório de conservação zoológico da Universidade Estadual de Londrina. Foram utilizados processos de destilação simples para aumentar o grau alcoólico e recuperação de formalina. Para a recuperação de etanol, destilação fraccionada, destilação azeotrópica e destilação extractiva foram utilizados. O resíduo foi destilado a partir de solução de formalina, e o formaldeído recolhido após a destilação foi quantificado através de titulação iodométrica, que provou ser um método muito eficiente e seguro. As amostras de etanol recolhidas foram medidas utilizando um densitômetro calibrado. Verificou-se que a concentração de formaldeído no resíduo era de 1,82% p / v e na solução recuperada era de 2,65% p / v. O grau máximo obtido na recuperação de etanol foi de 91,3 °INPM. Todos os processos de destilação foram eficientes na recuperação e purificação de solventes residuais, o que resulta numa diminuição no volume de resíduos e no custo de aquisição de novos reagentes. O novo projeto para uma escala simples e maior está em andamento para recuperar maiores volumes de formaldeído.

Palavras-chave: Formalina. Etanol. Destilação. Resíduos.

Abstract

This work presents developing processes for the recovery formalin and ethanol residues generated in laboratories. Disposal formaldehyde in unknown concentration and ethanol residue 70 °GL were collected in the zoological conservation laboratory. A simple distillation process for the purification and recovery formalin solution was used. Concerning ethanol recovery, the fractional process distillation, azeotropic distillation and extractive distillation were used. The quantification collected formalin residue and formaldehyde solution obtained after distillation was carried out by iodometry, which was found to be a very efficient and safe method. The alcoholic content collected ethanol samples was measured by using a calibrated densitometer. The formaldehyde concentration found in the residue was 1.82% m/v and in the recovered solution it was 2.65% m/v. The maximum alcoholic content obtained in the ethanol recovery was 91,3 °INPM, from tests performed on cars, the recovered ethanol showed signs being a efficient fuel, if approved after further testing may be reused in this function, in addition to being reused by the generator laboratory. All distillation processes have proved to be efficient in the recovery and purification this residues, resulting in a decrease in the volume residue and in the cost buying new reagents. The new project a larger scale simple distiller is in progress to recover higher amounts of formalin.

Keywords: Formalin, ethanol, distillation, residues

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Introduction

The steady growth the world population generated exacerbated industrialization process and urbanization resulting in major technological advances. Despite all the advances in the life quality in society, these processes most often occur at the expense the environment. The cause environmental harm generated by the society advance is arising mainly from industries that are major solid waste generators, chemical, biological and others, and also originated waste daily society activities (LIMBERGER, 2011; ALBERGUINI L. B. A.; SILVA, 2005).

The industrial progression sector is directly linked to the new process development, which most often originate in the research centers and universities. Consequently generating tailings in research and teaching activities in smaller volume than industrial effluents, but with damage to the environment in the same way (LIMBERGER, 2011). For many years the environmental issue has been overlooked by both the population and the government, but in recent years the environment has been very evident from the pollution effects on global warming, changes in climate, as water and food, and various other factors. With this environmental legislation establishing guidelines for issuing industrial effluents has become more rigid, forcing companies to treat their waste to reduce harm to the environment (FARAH, 2007).

In universities, mostly due to lack legislation and a supervisory body, waste management program is the absence management program generated in laboratories as a result is made improper disposal or specialized companies are contracted to collect the waste. So by financial and environmental issues is of very importance to waste treatment implementation in education centers (IARC, 1995).

Associating these waste management factors, the new process development and technologies for wastewater treatment, has grown rapidly. Chemical waste containing formaldehyde and ethanol are usually generated in large quantities in university laboratories, especially in centers of biological sciences, and most of them there is no proper disposal or treatment management or reuse these two wastes.

Formaldehyde is a reactive organic compound, has stabilizers, plasticizers and bactericidal properties, has application in several areas, is widely used as a fixing agent, embalming and conservation. In anatomy and histology laboratories are generated large formalin volumes residue in content approximately 3.7% w / v, this is because formalin solutions are renewed frequently and the generated volume becomes significantly greater. Because its toxic-

ity and its carcinogenic potential, improper disposal can cause various problems in the aquatic ecosystem where it is usually your final destination. With all these problems assigned, the reuse of waste containing formaldehyde is very important for the environment and for the university itself (FARAH, 2007; IARC, 1995; JARDIM, 1997)

Ethanol is a substance present in the daily lives of many laboratories, so the ethanol residues are generated in large quantities in the universities. The problem with this type of waste is not toxicity, since ethanol is used as fuel in automobiles, in foods, pharmaceuticals, alcoholic beverages, perfumes, and other consumer products directly by the company. The problem is that the cost for the removal of residual material, and local storage, however, the improper large disposal volumes may cause environmental problem (TUNGA, 2010)

Objectives

The objective of this work was the recovery of formaldehyde and ethyl alcohol used in anatomy laboratories.

Reuse process formaldehyde and ethanol

Waste management processes, as existing in universities, use common processes only treatment, waste inactivation in some tailings really can only neutralize the same, but the reuse of waste is more interesting both chemically and financially to the educational institution (IARC, 1995).

There are several inactivation methods or reduction of waste toxicity and depend on the present chemical compound, while for reuse processes the variety of processes is not extensive, these methods mostly involve the waste purification solutions resulting solution other than to be reused. The main operations for eliminating impurities are filtering and distillation. (LIMBERGER, 2011; IARC, 1995). In the case of ethanol, and formaldehyde residues filtration may be useful for removal of solid impurities. (LIMBERGER, 2011; SALE et al., 2008).

Distillation is a method of purification for liquid mixtures, is a process wherein a double physical state change, in which a substance, initially in the liquid state, is heated to the boiling temperature, turning into steam, and again cooled back to a liquid state (SALE et al., 2008; MCCABE; SMITH; HARRIOTTI, 2004). This is a process very old for purification, being very useful in waste recycling, as it involves the separation of liquids by difference in volatilities, many residues that are typically

aqueous mixtures of organic solvents can be distilled and reused later, various mixtures solvents may also be separated if boiling each temperatures significant they have a difference. Waste ethanol and formaldehyde are usually mixtures only in making water distillation a possible and viable process for purification and reuse (TUNGA, 2010; SALE et al., 2008; CHAGAS, 1999).

Azeotropes

In chemistry most studied concepts use an ideal model as a reference, in the case of liquid mixtures, the ideal model used is the law of Raoult, but the vast majority of liquid from vapor pressures do not follow this law, this violation of the law Raoult shown in the same form deviations. The deviations shown in Raoult's law can be positive or negative, and positive ones in which the vapor pressure of the mixture is greater than the pressure provided by the law and negative deviations are those where the vapor pressure of the mixture is smaller compared to the predicted the same. (CHAGAS, 1999; ATKINS P. W.; PAULA, 2008).

The deviations occurring in real slurries causes the formation of so-called azeotropic mixtures, having boiling at a constant temperature, like a pure substance, the composition and boiling temperature of these mixtures depends on the pressure (CHAGAS, 1999).

The positive deviation from Raoult's law means a boiling point of lower than expected mixing, that occurs in solutions that intermolecular forces of the mixture are weaker when compared to the solutions of the pure components. A mixture which has point of lower boiling point than of its components is called minimum-boiling azeotrope, so the components can not be separated by fractional distillation, the azeotropic mixture would come first in the column and the more volatile pure liquid, e.g. system of water and dioxane is a minimum boiling azeotrope, in which the water boiling temperature is 100 °C and dioxane 101,3°C and azeotrope boils at 87,7 °C equal to 0.476 mole fraction of dioxane. (ATKINS P. W.; PAULA, 2008; NATIONAL AGENCY OF PETROLEUM, 1991).

Extractive distillation and azeotropic distillation

Nowadays the techniques of mixture separation that exhibit azeotrope, are used more extractive distillation and azeotropic distillation, the most known application of these techniques is to obtain anhydrous ethanol. There are other more recent processes as dehydration molecular sieves and pervaporação, but the most widespread tech-

niques of distillation, since industries are mostly adapted for many years for this type of process (DIAS et al., 2009).

In both types of distillation, fractionation occurs after addition of a component to the mixture, which differs from the two techniques is the nature of the added component, the azeotropic distillation component is relatively volatile and extractive distillation component is relatively non-volatile (SALE et al., 2008).

In the azeotropic distillation of a binary mixture occurs forming a minimum boiling azeotrope with one of the blend components, then the azeotropic mixture passes to the distillate and the other component appears clean the bottoms product, the azeotropic mixture can then be separated in a decanting vessel and azeotropic agent reused. In the extractive distillation the extractive agent is not volatile and therefore not forming azeotropic mixtures it remains in the distillation feed liquid takes place, interacts more strongly with one of the components holding the same at the bottom of the column while the other component rises to the top column and appears in pure distilled (SALE et al., 2008; DIAS et al., 2009) azeotropic agent for extraction are chosen based on more strongly interact with components of the mixture an azeotropic agent can be added to a mixture in which components having boiling point near (low relative volatility) and different embodiments, the agent interacts more intensely with one of the components (for example, via hydrogen bridges), thus increasing the relative volatility, making the separation possible (SALE et al., 2008).

In 1950 was developed extractive distillation continuously for dehydration of ethanol using as the extractive agent is glycerol, which degradation problems and polymerizing the same, the process became exceeded. Currently in process, the glycerol was replaced by glycerin or ethylene, the latter being most commonly used (DIAS et al., 2009).

Together with extractive distillation was developed azeotropic distillation to obtain anhydrous ethanol with benzene and azeotropic agent, but due to its carcinogenic and uneconomical character in 1997 was banned its use in the process, as the industries were mostly adapted distillation azeotropic was only replaced by cyclohexane, and the process continues present application (DIAS et al., 2009).

Materials and methods

Collection of ethanol waste and Formaldehyde

Ethanol waste and formaldehyde were collected in the laboratory of Biological Sciences Center, where there is

a production of the two waste collected is about 10 L of formaldehyde disposal of unknown concentration and about 30 L of waste ethyl alcohol 70 °GL, used in the laboratory for fixation and conservation of zoological museum fish university, this collected amount is generated for a month in the laboratory. A random sample of paper was not necessary in this case because the entire amount waste generated has the same characteristics and concentration, has a homogeneous population hence the amount of collected waste is the waste generated throughout the laboratory.

Recovery process formaldehyde

The formaldehyde recovery was performed by distillation technique. The equipment consists of a round bottom distillation flask (Pyrex) 2 L a heating blanket (Fisatom), two metal grips two iron supports, balloon-condenser connector, thermometer, condenser coil, capacitor balloon connector receiver, 2 L round bottom flask receiver (Pyrex) and an elevator.

Were three distillations adding approximately 1 L of formaldehyde solution collected in the laboratory to the distillation flask, in the case of the presence of particulate matter in the solution was first performed the filtering thereof, after mounting the equipment was attached to the heater blanket and the cooling system water through the condenser. The formalin distillation process was performed at 96 °C (formalin boiling temperature) in the three replicates was expected to stop the process of distillation and collected to the distillate for subsequent quantitative analysis by iodometry formaldehyde concentration the same.

Quantitative analysis of formaldehyde

The iodometric titration was conducted according to USP Technologies.

All solutions, iodine, thiosulfate and other, were previously standardized.

Iodometric analysis for quantification of formaldehyde

Finally the obtained samples for quantification, a dilution was made in samples of the analytic reagents, as was expected a lower concentration of formaldehyde in the sample residue and liquid distillate. In the dilution of the three samples distillate and the collected residue was

transferred to 20 ml samples of 500 ml volumetric flasks were supplemented with distilled water.

Soon after dilution was performed the same iodometric titration procedure was done with the samples reagents. The iodometric titration of the samples were also done in triplicate, were analyzed to evaluate the distillate samples of the three distillations.

Ethanol recovery process

The recovery of ethanol was done by fractional distillation technique, Vigreux column, thermometer, ball condenser, receiver condenser balloon and balloon connector 2 L round bottom receiver

Successive distillations were carried fractionated using a Vigreux column of 30 cm, by adding approximately 1 L of ethanol residue in the distillation flask. After mounting the equipment connected to the heating blanket and the cooling system water through the condenser. The process of distillation of the alcohol solution was made to 78 °C (ethanol boiling temperature), after the distillation process, gauged to alcoholic obtained in the distillate using a hydrometer for mixing ethanol and water in a beaker 250 ml. The distillate was subjected to fractional distillation with another 30 cm Vigreux column, gauged to alcoholic with the densimeter again. The same procedure was carried out using a Vigreux column of 80 cm

Subsequently in order to increase the alcohol content of final distillate was added in 1.0 L distillation flask distillate obtained by the distillation process with Vigreux column of 80 cm with 50 ml of cyclohexane (Anidrol) causing the formation of an azeotropic mixture of cyclohexane and water, having a boiling point around 65 °C, then the azeotropic mixture has passed the distillate was collected and placed in a separatory funnel, so the azeotropic agent, in this case cyclohexane was separated and reused in other repetitions. After all output in the receiver flask cyclohexane, ethanol arrives as tail product and was collected and transferred to a 250 ml beaker to measure the alcoholic strength of the same using the densitometer. The azeotroping procedure was repeated three times using a 30 cmVigreux column.

Finally was added to the distillation flask, 1L distillate obtained by the distillation process with 80 cmVigreux column with 50 ml of ethylene glycol (anhydrous), with the function of fixing the water to the distillation flask, distillation was done normally. The distillate was transferred to a 250 ml beaker for measuring the alcohol content therein using the densitometer. Extractive distillation was repeated three times using a 30 cmVigreux column.

To evaluate the efficiency of Vigreux column with 30 cm high, was added to the distillation flask, 500 ml of distilled water and 500 ml of ethyl alcohol 93,3°INPM, it carried out the fractional distillation process is collected and the- distilled to measure the alcoholic content. All distillation processes performed were made with a water reuse system used to cool the condenser and then condensing the vapor. The condenser outlet water was collected in a recipient of 100 L, which by a system of hydraulic pumps is directed to the water box on the laboratory, at this time it is cooled naturally and back to the supply record that carries water the condenser.

Results and discussions

Formaldehyde recovery process

Formaldehyde residue generated at the collection point is due to the constant renewal of formalin solution used for fixation of biological tissues, in the case of laboratory fish are fixed with formaldehyde solution 3.7% w / v and then kept in ethyl alcohol 70°GL, the residue generated presented up with a yellowish tinge due to the presence of fats, proteins and even fish from dirt, since they are collected from the natural environment and are immediately placed in formalin solution for and immediate fixation and preservation of tissues until the arrival to the laboratory, where the collected animal is transferred to alcoholic solution. After performing the simple distillation process, the appearance of the solutions have changed, from turbid and yellow to completely clear and translucent, showing a sign of purification performed in distillation.

Formaldehyde quantitative analysis

The analysis of the formaldehyde solution by iodometry method proved very efficient for quantization in aqueous solution, as evidenced in the measurement method using the two samples of commercial formaldehyde 37% w / v. For PA formaldehyde (Fmaia) with certified concentration on the label of 36-38% m / v, the method proved to be quite accurate obtaining a concentration of 37.09% w / v (value obtained from the average of the three repetitions of method) to another sample Formaldehyde PA (Alphatec) with concentration of 36.5 to 38% w / v, the method resulted in an average concentration of 36.88% w / v. The concentrations obtained show very consistent results, within the concentration ranges specified in the labeling, showing that the technique used for quantification can be safely used to determine the sample residue

and formaldehyde distillate, being more efficient, accurate and applicable than methods existing testing kits on the market.

The concentrations of formaldehyde in the distillate and residue collected are shown in Tables 3 and 4.

Table 1 – Formaldehyde concentration in the collected waste.

Titration	Formaldehyde concentration (% m/v)
1	1.90
2	1.74
3	1.82
Average	1.82

Source: by the author.

Table 2 – Formaldehyde concentration in the distillate.

Tritation	1 Concen- tration (% m/v)	2 Concen- tration (% m/v)	3 Concen- tration (% m/v)
1	2.67	2.61	2.61
2	2.60	2.69	2.69
3	2.60	2.69	2.69
Average	2.62	2.66	2.66

Source: by the author

Before the experiment is expected that after the distillation of formaldehyde was obtained at concentrations around 3.7% w / v, but the initial concentration of formaldehyde at residue was lower than expected, the deterioration of formalin residue shewed to be more intense, starting from a 1.82% solution w / v, it has not been possible to achieve values around 3.7% w / v, but recovered with formalin concentration of 2.66% w / v (mean the three distillations) will be reused by the laboratory by adding it to the dilutions and consequently reducing the cost to purchase new reagents.

Thus the implementation of a still large scale for the recovery of large volumes of formalin, it becomes important and viable for a larger waste treatment capacity, since at the University occurs generation of waste formaldehyde in large quantities in other laboratories beyond which was used for collection of waste in the experiments. The generated savings is great, because there will be reduced purchase of reagents and there will be costs for the removal of waste by specialist companies. The system for

recovery on a larger scale, is being planned for implementation with a treatment capacity of approximately 500 L by distillation of formaldehyde.

Ethanol recovery process

The alcohol residue generated in the laboratory is due to the constant renewal of the ethanol solution used for the conservation of biological tissues, in this case fish are fixed with formaldehyde solution 3.7% w / v and later preserved in ethyl alcohol 70 °GL ie 64.8 °INPM. INPM degree indicates the fraction mass of alcohol in the mix [12]. The residue collected presented with ameralada staining, as well as the formaldehyde, although the ethanol has the function to retain the tissue and the characteristics of the stored body with time the solution turns removing animal proteins and fats, becoming cloudy solution yellowish fact makes it difficult to view the body inside the storage container, and causes the exchange solution, the alcoholic generating waste.

After completion of the fractional distillation process, the appearance of the solutions have changed, from turbid and yellow to completely clear and translucent, showing evidence of the purification carried out in distillation.

In fractional distillation using a Vigreux 30 cm column , the concentration of the alcoholic solution obtained by measuring with the densimeter was 84,2°INPM, while the distillate obtained in the fractional distillation performed with Vigreux column 80 cm high concentration obtained was 86,0° INPM.

The result shows the influence of the height of the column at the alcohol obtained in the distillate theoretically higher the number of theoretical plates in a more efficient column it is in the separation of the mixture components, the higher the concentration of the distillate, but the Vigreux columns used in the experiment did not really have such dishes as industrial columns that provide the contact between the ascending vapor and descending liquid, but the walls have recesses to increase this contact, the greater the contact surface between the ascending phase (steam) and the descending phase (fluid) the greater the efficiency separation, as expected a higher concentration of the distillate in the distillation 80 cm column which has a larger contact surface than with the 30 cm column, then the result was consistent with expectations, but with a small difference between the concentrations given the large difference in height of the columns. In fractional distillation of the distillate obtained from the distillatio30 cm column , using the same column was obtained alcohol content of 91.1 °INPM in the same process carried out with

the 80 cm column graduation achieved was 91.1 °INPM. It was expected by theory a higher alcoholic content in the procedure with 80 cm column , but the result was the same in both columns, this can be explained by the fact that the mixture of ethanol and water having a minimum boiling azeotrope at 93° INPM of ethanol which is equal to a molar ethanol fraction 0.89, the azeotrope reach this point the concentration of ethanol in liquid phase and the vapor phase is exactly the same, so the complete separation does not occur and obtaining pure ethanol (anhydrous) in a conventional distillation becomes unviable, or near that point because the concentrations in the two phases were very similar separation becomes harder, the ethanol-water mixture at this concentration boiling before absolute ethanol so the distillate was no difference in concentration of redistillation, comparing the two columns.

For hydrous ethanol can see sold as fuel in Brazil he must have alcohol within the limits established by the National Petroleum Agency (ANP), and this range of alcohol content from 92.5 to 93.8 °INPM, with conventional fractionated distillations performed, obtained a maximum rating of 91.1 °INPM, so in an attempt to increase the alcohol content obtained, there was the azeotropic distillation process with cyclohexane and extractive distillation with ethylene glycol, the result was an alcohol content 91.3 °INPM, the variation in the alcohol content was very low compared with conventional distillation, the lower result may be caused by the absence of a reflux system the fractionated distillation performed also technically two distillations are essentially industrial processes continuously and extractive and azeotropic fractional distillation ethylene glycol and cyclohexane are added in the distillation system from the top of the column, all these process variations may have caused the low yield of the result.

The results show the same 30 cm and 80 cm column efficiency redistillation thus realized the efficiency of the column test using a 30 cm column , starting from a mixture of 500 ml of distilled water and 500 ml of ethanol the 93,3° INPM, or a water-alcohol mixture with 46,65°INPM, gave a distillate with undergraduate 86,0°INPM.essas process variations may have caused the low yield of the result.

The ethanol obtained through two consecutive distillations with 91.1 °INPM can be reused by the generator laboratory very extensively residue, is for the same function in preserving fish in this case a dilution is required, or for other functions such as cleaning and etc.

Regarding the use as automotive fuel, ethanol obtained can not be marketed, since it is not appropriate within the parameters established by the ANP, but in tests with cars powered by ethanol in vehicles, ethanol samples to

91.1 and °INPM 91.3 °INPM apparently were efficient in the operation of the engine, obtaining evidence of the possibility of its use as fuel. More extensive tests should be performed with ethanol recovered to verify the possibility of its use in vehicles and can generate more that advantage and economy, besides the generated waste reduction and the purchase of new reagents.

Conclusion

Through distillation Vigreux column it was possible to obtain a clear and translucent formalin solution, not in sufficient concentration for direct reuse the same function, but the recovered solution can be added at dilutions carried out in the laboratory, reducing waste volume generated and purchase of new reagents. Demonstrating the feasibility of implementing a still larger scale, to recover higher formaldehyde volumes generated in laboratories.

The iodometric method proved to be very effective in determining the concentration of aqueous solutions of formaldehyde, providing security for quantification of samples.

Fractional distillation of the alcohol residue with a 30 cm and 80 cm Vigreux column showed the same results when performed twice in sequence, the azeotropic and extractive distillation obtained alcohol content was very close to conventional distillation, with the obtained ethanol concentration reuse of ethyl alcohol by the generator laboratory is completely feasible.

In tests conducted in the university car, there are indications that the obtained ethanol can be used as fuel, but regulated tests should be performed to find the possibility of use.

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