PRETREATMENT OF INSTANT COFFEE WASTEWATER BY COAGULATION AND FLOCCULATION

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Dedicated to my beloved parents…

ACKNOWLEDGEMENTS

The author would like to express his sincere appreciation and gratitude to his supervisor, Dr. Mohd. Ariffin Bin Abu Hassan, of Department of Chemical Engineering, Faculty of Chemical and Natural Resources Engineering, Universiti Teknologi Malaysia (UTM) for his advice, guidance, support and encouragement throughout his research study.

Special thanks to the staffs of the Faculty of Chemical and Natural Resources Engineering, Universiti Teknologi Malaysia (UTM), especially the technician and lab assistants for their assistance and cooperation in carrying out the experimental studies. The author also appreciated En. Zukarnai from M/s. Dan Kaffe (M) Sdn Bhd for his cooperation in completing the study.

The author also like to express his grateful to the course-mates for their helps and supports in this study. At the last but not least, the encouragement, support and love from parents and family members are sincerely acknowledged.

ABSTRACT

This study investigated the performance and effectiveness of coagulation and flocculation by using aluminum sulfate, ferric chloride and chitosan as a pretreatment for instant coffee wastewater. The removal of total suspended solids (TSS), turbidity, chemical oxygen demand (COD) and color by jar tests were used to determine the optimum dosage and pH. Optimum conditions for aluminum sulfate and ferric chloride was 1000 mg/L at pH 7, while for chitosan was 100 mg/L at pH 6. The dosage of chitosan was 10 times much lesser as it had the higher charge density. Chitosan exhibited the best result for turbidity and TSS removal by 96.95% and 91.43%, respectively. This was followed by ferric chloride that removed 95.38% turbidity and 91.43% TSS; and aluminum sulfate with 87.65% turbidity and 88.57% TSS removal. At the same time, ferric chloride was the best coagulant for color and COD removal, with 95% and 66.45%, respectively. This was followed by the aluminum sulfate with 90% color and 56% COD removal; and chitosan that removed 88.55% color and 46.46% COD. Chitosan produced the faster aggregation of colloids, with the lowest volume of sludge of 60 mL. This was followed by aluminum sulfate with 87 mL and ferric chloride with 103 mL. The cost of ferric chloride using per day, RM20,340 was the lowest, which was 16.8% lower than aluminum sulfate and 119.8% lower than chitosan. Overall, all coagulants can be used for pretreatment of coffee wastewater, with ferric chloride was preferable.

ABSTRAK

Kajian ini mengkaji prestasi dan keberkesanan koagulasi dan flokulasi dengan menggunakan aluminum sulfide, ferric chloride dan kitosan sebagai rawatan awal untuk air sisa kilang kopi segera. Pengurangan jumlah pepejal terampai (TSS), kekeruhan, warna dan keperluan oksigen kimia (COD) melalui ujian balang telah digunakan untuk menentukan dos dan pH optimal. Keadaan optimal bagi aluminum sulfate dan ferric chloride adalah 1000 mg/L dengan pH 7, manakala untuk kitosan adalah 100 mg/L dengan pH 6. Dos yang diperlukan untuk kitosan adalah 10 kali ganda lebih rendah kerana kitosan mempunyai ketumpatan cas yang lebih tinggi. Kitosan menunjukkan keputusan yang terbaik untuk pengurangan kekeruhan dan TSS dengan pengurangan 96.95% dan 91.43% masing-masing. Ini diikuti dengan ferric chloride yang mengurangkan 95.38% kekeruhan dan 91.43% TSS; dan aluminum sulfate dengan pengurangan 87.65% kekeruhan dan 88.57% TSS. Pada masa yang sama, ferric chloride merupakan agen koagulasi terbaik untuk pengurangan warna dan COD, dengan nilai 95% dan 66.45%. Ini diikuti dengan aluminum sulfate dengan pengurangan 90% warna dan 56% COD; dan kitosan yang mengurangkan 88.55% warna dan 46.46% COD. Kitosan menghasilkan penggumpalan pepejal yang paling cepat, dengan isipadu enapan sebanyak 60 mL. Ini diikuti dengan aluminum sulfate pada 87 mL dan ferric chloride pada 103 mL. Kos ferric chloride yang digunakan sehari, RM20,340 adalah yang terendah, dengan 16.8% lebih rendah daripada aluminum sulfate dan 119.8% daripada kitosan. Keseluruhannya, semua agen koagulasi berkeupayaan digunakan sebagai rawatan awal untuk air sisa kopi, dengan ferric chloride adalah terpilih.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

Al^{3+}	-	Aluminum ion
$Al(H_2O)_6^{3+}$	-	Aluminum aquometal complexes
$Al(OH)^{2+}$	-	Cationic mononuclear aluminum species
$Al(OH)_2^{+}$	-	Cationic mononuclear aluminum species
$Al(OH)_3$	-	Aluminum hydroxide
$Al(OH)_4^{-}$	-	Aluminate ion
$Al_2(SO_4)_3$	-	Aluminum sulfate
$Al_2(SO_4)_3.14H_2O$	-	Aluminum sulfate 14 hydrate
$Al_2(SO_4)_3.16H_2O$	-	Aluminum sulfate 16 hydrate
$Al_2(SO_4)_3.18H_2O$	-	Aluminum sulfate 18 hydrate
$Al_2(OH)_2^{4+}$	-	Cationic polynuclear aluminum species
$Al_6(OH)_{15}^{+3}$	-	Cationic polynuclear aluminum species
$Al_7(OH)_{17}^{+4}$	-	Cationic polynuclear aluminum species
$Al_8(OH)_{20}^{+4}$	-	Cationic polynuclear aluminum species
$Al_{13}(OH)_{34}^{5+}$	-	Cationic polynuclear aluminum species
BOD	-	Biochemical oxygen demand
^{o}C	-	Degree celsius
$Ca(HCO_3)_2$	-	Calcium bicarbonate
CaOH	-	Calcium hydroxide
$CaSO_4$	-	Calcium sulfate
C_i	-	Initial concentration
C_f	-	Final concentration
cm	-	Centimeter
CO_2	-	Carbon dioxide
COD	-	Chemical oxygen demand

$-COO^-$	-	Anionically charged carboxyl group
-COOH	-	Carboxyl groups
d	-	Thickness of the layer surrounding the shear surface
D	-	Dielectric constant of the liquid
DA	-	Degree of acetylation
DLVO	-	Derjagin-Landau-Vervey-Overbeck theory
DD	-	Degree of deacetylation
DO	-	Dissolved Oxygen
DOE	-	Department of Environment
EQA	-	Environmental Quality Act 1974
Fe^{3+}	-	Ferric ion
$Fe(OH)_3$	-	Ferric hydroxide
$Fe_2(SO_4)_3$	-	Ferric sulfate
$Fe(OH)_2^+$	-	Cationic polynuclear ferric species
$Fe_2(OH)_2^{+4}$	-	Cationic polynuclear ferric species
$Fe_3(OH)_4^{+5}$	-	Cationic polynuclear ferric species
$FeCl_3$	-	Ferric chloride
$FeCl_3{\cdot}6H_2O$	-	Ferric chloride 6 hydrate
g	-	Gram
g/mol	-	Gram per mole
g/cm^3	-	Gram per centimeter cubic
HAc	-	Acetic acid
HCl	-	Hydrochloric acid
H_2O	-	Water
H_2SO_4	-	Sulfuric acid
H_3O^+	-	Ion hydrogen
ICO	-	International Coffee Organization
kg	-	Kilogram
kg/m^3	-	Kilogram per meter cubic
L	-	Liter
m^3/day	-	Meter cubic per day
mg	-	Miligram
mg/L	-	Miligram per liter
mg/mL	-	Miligram per mililiter

mL	-	Mililiter
M	-	Molar
NaOH	-	Sodium hydroxide
$-NH_2$	-	Amino group
$-NH_3^+$	-	Positively charged amino group
NTU	-	Nephelometric Turbidity Units
-OH	-	Hydroxyl ion
PtCo	-	Platinum-cobalt
Q	-	Flowrate
q	-	Charge per unit area
RM	-	Ringgit Malaysia
rpm	-	Rotation per minute
Si^{4+}	-	Silicon ion
TSS	-	Total suspended solid
UASB	-	Upflow Anaerobic Sludge Blanket
μm	-	Micron
W_i	-	Initial weight
W_f	-	Final weight
ζ	-	Zeta potential

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Introduction

Nowadays, coffee is one of the most popular commodities in the developed world. Supermarkets and coffee shops are always stocked with a plenty supply of coffee blends and flavors. In the past, the fresh coffee for home and catering brewing is available by the method of filtration (percolation), infusion or boiling (decoction) the roast and ground coffee (Clarke and Macrae, 1987). The process will normally take time with the requirement of some household or catering-type equipment. With the desire to make coffee instantly by simply mixing a dry or liquid concentrate with hot water, the product of soluble or instant coffee is marketed worldwide through the improvement of modern processing technology in food and beverage industry.

Instant or soluble coffee is a beverage that derived from brewed coffee beans. The coffee is dehydrated into the form of granules or powder through various manufacturing processes. It can be rehydrated using hot water to provide a drink similar to brewed coffee. Besides, the instant coffee is also available in the form of concentrated liquid. Instant coffee is manufactured from coffee beans through a series of process, including roasting, grinding, extraction, concentration, drying and packing (Clarke and Macrae, 1987). It is normally to produce a soluble powder or granules, either by spray-drying or freeze-drying technique.

In recent years, the growth of the instant coffee market due to the modern consumer's desire for a convenience product has been impressive. Consequently, the market expansion has led to the production of increasing quantities of wastewater with high pollution potential and the spent coffee grounds as an unwanted by-product of instant coffee manufacture (Clarke and Macrae, 1987). Many research studies have been conducted for the treatment, reusing and recycling of spent coffee grounds but there is lack of the focus on the instant coffee processing wastewater. The wastewater of instant coffee industry is primarily generated from cleaning operations including equipment cleaning and floor washing. It is important to treat the instant coffee wastewater with proper, effective and economical practices prior to their discharge into the receiving water.

1.2 Research Background

The first soluble instant coffee was invented by Dr. Satori Kato, a Japanese chemist in Chicago, and the soluble coffee was first sold to the public at the Pan-America Exposition of 1901 (Wrigley, 1988). Shortly thereafter, George Washington developed his own instant coffee process and it was first appeared on the American market in 1910 (Ukers, 1935). The Nescafé brand that introduced a more advanced coffee refining process was launched in 1938 (Clarke and Macrae, 1985). On a global basis, it is estimated that in 1980 about 19% of coffee consumed went into soluble manufacture and today the countries with the highest total consumption of instant coffee are, in order, United States of America (USA), United of Kingdom (UK), Japan, France, Germany and Canada (Clarke and Macrae, 1985). Now, the instant coffee can be found all over the world and it has been widely used for decades.

Instant coffee comes in three forms: freeze-dried, spray-dried and liquid coffee extract. A good cup of coffee can be made from the instant coffee with a number of advantages over fresh brewed coffee. The instant coffee allows the consumer to make coffee in an ease and convenience way without any equipment other than a cup and without having to discard any damp grounds. The preparation of coffee is simple and fast as no time is required for infusing the coffee and it is ready as soon as the hot water is added. The quality of instant coffee also has grown dramatically over the years. It stays fresher longer and has long shelf life because natural coffee, especially in ground form, loses flavor as its essential oils evaporate over time. In short, the instant coffee is fast, cheap and clean.

Instant coffee has become a product that attracts great attention in the food and beverage industries of Malaysia. Although Malaysia is not the major country of coffee plantation and green coffee production, there are more than thirty soluble and instant coffee manufacturers all around the country. The high technologies are used to manufacture regular and agglomerated instant coffee in the form of powder or granules as well as canned liquid coffee. The rapid growth of instant coffee industry is accompanied by a staggering increase in the amount of wastewater produced. The major sources of wastewater produced in the instant coffee processing industry include the water used for the cleaning of extractor, spray dryer, freeze concentrator, separator, heat exchanger, boiler, evaporator finisher and pasteurizer, washing the floors and working areas (Lim, 1999).

The International Coffee Organization (ICO) has proposed the Common Code for the Coffee Community (4C) to create a common global code to cover the economic, social, and environmental pillars for achieving greater sustainability of development for coffee industry (Osorio, 2005). From the aspect of environment that related to coffee processing, there are three main issues to be considered, which included proper wastewater treatment, utilizing by-products, and conserving energy. The water quality and aquatic ecosystem will be affected seriously if the high strength and polluted wastewater from coffee processing is discharging into the receiving water without any suitable treatment system.

Different products of instant coffee with different technology can lead to different amounts and quality of wastewater produced. The production of instant coffee gives rise to substantial volumes of wastewaters containing a wide variety of pollutants. In general, the wastewaters contain higher value of biochemical oxygen demand (BOD), chemical oxygen demand (COD), total suspended solid (TSS) and turbidity. The wastewaters also possess a distinctive dark brown color. The pH can be in a wide range depending of its sources. Coffee wastewater contains high organic loads which may result in dissolved oxygen depletion in the receiving waters (Ricardo, 1996). The volume, concentration and composition of the effluent arising in the manufacturing plant are dependent on the type of product being processed, the production program, operating methods, design of the processing plant, the design of water management being applied, and subsequently the amount of water being conserved (Lawrence *et al*., 2004).

For instant coffee industry, the wastewater is normally treated by physical and chemical, biological processes. The pretreatment or primary treatment is a series of physical and chemical operations, which precondition the wastewater as well as remove some of the wastes. The treatment is normally arranged in the sequence of screening, flow equalization, coagulation-flocculation, sedimentation, and dissolved air flotation (Lawrence *et al*., 2004). Screening is applied to remove coarse particles in the influent while flow equalization is a method used to overcome the operational problems caused by flowrate variations. Coagulation is used to destabilize the stable suspended solids and colloidal particles while flocculation is used to aggregate the destabilized particles to form a larger and rapid-settling floc. This normally acts as preconditioning process for sedimentation and / or dissolved air flotation.

Biological processes have been developed for secondary treatment system to remove the dissolved and particulate biodegradable components in the wastewater (Metcalf and Eddy, 2003). Microorganisms are used to decompose the organic wastes. With regard to different growth types, biological systems can be classified as suspended growth or attached growth system. Furthermore, it can also be classified by oxygen utilization: aerobic, anaerobic and facultative. A research study of anaerobic treatment by upflow anaerobic sludge blanket (UASB) process was carried out by the Nestlé Foods Corporation Purchase, which has one of the largest freeze-dried coffee plant located in Freehold, New Jersey and generated approximately 760 m^3/day of wastewater (Lanting *et al*., 1988). They study the treatability of coffee wastewater by using four pilot systems under both mesophilic and thermophilic conditions. The COD removals were between 49 to 69%.

1.3 Problem Statements

There has been limited research on coagulation and flocculation process for the pretreatment of instant coffee wastewater as comparing with other food and beverage industries. Due to the high concentration of organic pollutants and suspended solids in wastewater of soluble coffee processing, its disposal without an appropriate treatment into the receiving water has become undesirable because it will be very dangerous for the water bodies and human health. The wastewater discharged from any industries in Malaysia must follow the stringent effluent standard of Environment Quality Act (EQA) 1974. Thus, a proper and effective treatment system is needed. The pretreatment systems such as coagulation and flocculation processes play a significant role for overall performance of the wastewater treatment plant. It is important in reducing most of the suspended solids and organic matter in the raw wastewater before entering into the secondary treatment system (Metcalf and Eddy, 2003).

The most widely used coagulants in wastewater treatment are inorganic metal salts, such as aluminum or iron salts. Aluminum sulfate and ferric chloride have been extensively used as a primary coagulant in wastewater treatment. This is due to their effectiveness, cheap, easy to handle and availability (Edzwald, 1993). However, its best performance and cost-effectiveness can only be achieved during the optimum conditions of coagulation and flocculation process. It is important not to overdose the coagulants because a complete charge reversal and restabilization of colloid complex can be occurred.

Recently, there is more attention on the extensive use of aluminum-based coagulant. Besides producing large amount of sludge, the high level of aluminum residual in the treated water has raised concern on public health. McLachlan (1995) discovered that intake of large quantity of alum salt may cause Alzheimer disease. To minimize the detrimental effect accompanied with the use of alum, polymers are added either with alum or alone and have gradually gained popularity in water treatment process. Synthetic polyelectrolytes produce sludge of better dewatering characteristics with smaller volume and facilitate better filtration, but their long-term effects on human health are not well understood (Pan *et al*., 1999). Furthermore, the sludge formed during flocculation with synthetic polymers has a limited potential for recycling due to the non-biodegradability of synthetic polymers (Bratskaya *et al*., 2004).

Therefore, it is necessary to develop a more effective and environment friendly coagulant as a viable alternative to these chemical coagulants. Since most of the wastewater colloids are negatively charged, natural cationic polyelectrolytes, such as chitosan has become a particular interest. Besides promotes an excellent pollutant removal, the biodegradability and non-toxic nature of chitosan provides an opportunity for water recycling in the industry and sludge recovery in the production of fertilizers or additives for animal feeding mixture. However, chitosan is same as aluminum sulfate or ferric chloride that must be applied in the optimum coagulation and flocculation conditions for the best performance and cost-effectiveness.

1.4 Objectives

The objectives of this study are stated as below:

1. To investigate the efficiency of coagulation and flocculation processes as a pretreatment for coffee industrial wastewater by using different types of coagulants (aluminum sulfate, ferric chloride and chitosan).

2. To evaluate the performance of aluminum sulfate, ferric chloride and chitosan in the reduction of total suspended solids (TSS), turbidity, chemical oxygen demand (COD) and color.

3. To determine the optimum conditions for the coagulation and flocculation processes of abovementioned coagulants.

1.5 Scope of Works

The steps and scopes leading to the objectives were:

1. To study and to determine the characteristics of raw wastewater from instant coffee processing industry.

2. To investigate the optimum dosage of the selected coagulants in reducing the pollutant load.

3. To determine the optimum pH for the coagulation and flocculation processes.

4. To compare the treatment efficiency by using aluminum sulfate, ferric chloride and chitosan.

1.6 Contribution of Study

This research will contribute on providing the optimum conditions of coagulation and flocculation process for common coagulant such as aluminum sulfate and ferric chlorite, applying for instant coffee processing wastewater. The research also explores the potential of coagulation treatment system by using chitosan, a natural polyelectrolyte. The knowledge obtained from this research will allow more efficient, effective and economical design and operation of pretreatment process for instant coffee industrial wastewater by using coagulation and flocculation.

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