Removal of Color Substances in Molasses Wastewater by Combined Biological and Chemical Processes

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Abstract

In this study, we found that the usual biological treatment processes, especially anaerobic pond system, could easily reduce chemical impurities such as COD, BOD_5 and so on from molasses wastewater (MWW). The BOD_5 and COD concentration in stillage of Sang som alcohol distillery factory were reduced by about 90% and 46%, respectively by 67,200 M^3 anaerobic pond (HRT as 220 days). But the color substances could not be removed by above treatment system. The color intensity (as the optical density at 475 nm) of the stillage and treated stillage from the anaerobic pond (An-MWW) were 84.4 units and 78.2 units, respectively. But the molecular weight distribution patterns of color substances in stillage and An-MWW were not the same. The stillage contained the larger molecular weight pigments while the An-MWW contained the smaller molecular weight pigments. The suitable coagulants for removing color substances from stillage and An-MWW were CaO and $FeCl_3$, respectively. The pretreatment of the MWW by aeration did not show any change in the color intensity and molecular weight of color substances. But for the pretreatment of stillage and An-MWW by aeration with sludge-added, the color intensity of MWW decreased by about 45.14% and 9.85%, respectively. However, the pretreatment of MWW by both aeration with and without sludge-added gave benefit for color removal efficiency in the chemical coagulation step. The concentration of the coagulants (CaO for stillage and $FeCl_3$ for An-MWW) which were used for removing the color substances from pretreated MWW were lower than from raw MWW. The concentration of CaO which was used for removal of color substances in stillage and stillage which was pretreated by aeration with and without sludge-added was 83.0 g/L, 60.0 g/L and 50.0 g/L, respectively. And the concentration of $FeCl_3$ which was used for removal of color substances in An-MWW and An-MWWs which were pretreated by aeration with and without sludge-added was 29.0 g/L, 20.0 g/L and 12.5 g/L, respectively. The removal efficiencies for stillage and An-MWW which were pretreated by aeration with sludge-added for 96 hrs by suitable coagulants (CaO for stillage and FeCl3 for An-MWW) were 95.25% and 99.65%, respectively.

From the above results, we can say that color substances in both types of MWWs could be removed by simple coagulants (CaO for stillage and $FeCl_3$ for An-MWW). But, if we would like to increase the color removal efficiency in the chemical coagulation step, pretreatment of MWW by aeration with or without sludge-added is necessary.

For the application, the MWW had to be pretreated by aeration with or without sludge-added for 96 hrs. Then, the supernatants of the pretreated MWWs were coagulated by suitable coagulants ($FeCl_3$ for An-MWW and CaO for stillage). After that, the supernatants of the reaction mixtures were treated by conventional biological treatment processes such as oxidation pond or aerated lagoon for removal of the remaining impurities. The quality of treated wastewater from our recommended process would pass the standard requirement of Department of Industrial Works of Thailand.

Key words: Molasses wastewater, Stillage, Sludge, Aeration, Coagulation, CaO, $FeCl_3$, Alum.

1. Introduction

Molasses is the most important by-product from cane sugar factories [1,2] because molasses has a high commercial value due to usefulness as a carbon source for fermentation industries, a bio-fertilizer and a feed for domestic animals [3]. However, the use of molasses as a raw material for fermentation industries has the disadvantage that a large amount of color substances remain in the fermentation residue after recovery of the products. The main color substance, melanoidin, can not be decomposed by the usual biological treatment processes [4,5] and they keep the high level of COD value.

In Thailand, all of the alcohol producing factories use molasses as the raw material and discharged molasses wastewater (MWW) is about 10 times of alcohol produced. Several processes [6,7,8] are used for treating the MWW such as biological treatment process (anaerobic and aerobic treatment processes). MWW is used as the aqua-life feed and bio-fertilizer. However, the use of MWW as the feed of aqua-life did not play from the economical point of view due to the small volume used. Also the quality of the bio-fertilizer used is not so high in comparison with other bio-fertilizer or chemical fertilizer (It might be same as soil conditioner). Several alcohol factories attempted to treat MWW by anaerobic methods such as methane fermentation, anaerobic pond or facultative pond followed by aerobic treatment such as activated sludge, aerated lagoon or oxidation pond [10]. But, using the above biological treatment processes, the color of the MWW still remained. And the COD content of treated wastewater was still higher than the standard permission value of the Ministry of Industry, Thailand [11], Nowadays, the chemical processes such as chemical precipitation, chemical adsorption or adsorption by activated carbon are used for removal of the color substances from the biological treated molasses wastewater. But the color removal efficiency by above processes still has disadvantages due to high operation cost, high consumption of chemical agents, fluctuation of the color removal efficiency and the large volume of solid waste produced.

Under the situation mentioned above, we would like to check the kinds of molasses wastewater (treated or untreated molasses wastewater) which are suitable for simple chemical coagulation processes. The suitable chemical agent and the optimal conditions for above chemical coagulation process were investigated. Furthermore, the pretreatment by biological processes was applied for improving the removal efficiency for the chemical coagulation step for the purpose of removing color substances, COD, BOD_5 and so on with highest efficiency.

2. Materials and Methods

Molasses wastewater: The molasses wastewater from wastewater treatment plant of Sang som alcohol distillery factory, which were collected for using in this study, were stillage and treated stillage from anaerobic pond (An-MWW)

Jar test: The wastewater samples, which were used in this study, were stillage and An-MWW of Sang som alcohol distillery factory. The experiments were done in three steps by using 3 kinds of coagulants: alum, $FeCl_3$ and CaO as follows.

First experiment was determination of suitable chemical agents for coagulation of color substances from the stillage and An-MWW. Three kinds of chemical agents, alum, $FeCl_3$ and CaO were used in this experiment. 200 ml of wastewater sample in 500 ml beaker was adjusted to pH 6 by 20% sodium hydroxide solution. Then, 4 mg of chemical agent (alum, $FeCl_3$ or CaO) was added and rapidly mixed (100 rpm) for 3 min and slow mixed (30 rpm) for 12 min, respectively. Then, the supernatants were collected for determination of color intensity after sedimentation for 30 min.

Secondary experiment was determination of the optimal pH for coagulation. A 500 ml of wastewater sample in 1,000 ml beaker was adjusted to pH 4, 5, 6, 7, 8 and 9, respectively by 20% sodium hydroxide solution or 20% sulfuric acid solution. Then, 4 mg of suitable chemical agent which was selected from the first experiment, was added to each beaker with fast mixing (100 rpm) for 3 min and slow mixing (30 rpm) for 12 min, respectively. The supernatant was collected after sedimentation for 30 min for color intensity determination.

Third experiment was determination of optimal concentration of coagulants. A 500 ml of wastewater sample in 1,000 ml beaker was adjusted to optimal pH which was selected from the secondary experiment. Then the various concentrations of coagulants were added to each beaker with fast mixing for 3 min and slow mixing for 12 min, respectively. The supernatants were collected after sedimentation for 30 min for determination of color intensity.

Pretreatment by biological processes: The experiments were done in 2 sets of 10 liter reactors. 5 liters of stillage or An-MWW was put in each reactor. 10,000 mg of sludge (dry weight) from Bangkok municipal waste water treatment plant (Siphaya branch) was added to first reactor. Then, both reactors were fully aerated for 4 days (96hrs). During aeration, the supernatants were collected for determination of chemical properties and color intensity. The supernatants from both reactors were also collected for use as the pretreated MWW solutions to check the color removal efficiencies in the chemical coagulation step.

Gel filtration chromatography: Determination of molecular weight patterns of stillage and An-MWW solutions and their treated solutions by Gel filtration chromatography method were done. The MWW solutions and their pretreated solutions were placed on a column of Sephadex G-50 (2.6x95 cm), previously equilibrated with 0.01 M phosphate buffer (pH 5.0) and eluted with same buffer at a flow rate of 0.25 ml/min. The effluent was collected at 5 ml/tube. The color intensity of each fraction was measured at 475 nm as mentioned below.

Assay of the chemical compositions of the waste water : The BOD_5, COD, TKN, Total phosphorus and SV_{30} were determined by standard method of water and wastewater analysis[12)]

Estimation of the color intensity of the waste water and its removal yield: The sample was diluted with 0.1 M acetate buffer solution pH 6.0 after being centrifuged at 6,000 Xg for 15 min and the color intensity of the diluted solution was measured at 475 nm by a spectrophotometer (LKB, model Biochrom Ultra-space2, England). The percentage of color removal was calculated as the color intensity of the waste water treated against that of original wastewater. The removal yield was expressed as the degree of decrease in the absorbency at 475 nm against the initial absorbency at the same wavelength.

3. Results

Chemical compositions of molasses wastewaters: The wastewater treatment plant of Sang som alcohol distillery factory is located at Ampoe Dontoom Nakhonphatom province. The system is the anaerobic pond system. It consists of one big pond. The size of the anaerobic pond is 11,200 M^2 in surface area and 6 M in deep. The total volume of anaerobic pond is 67,200 M^3. The stillage produced is about 300 M^3/day. The HRT of the system is 220 days. The chemical compositions of the stillage and An-MWW are shown in table1. The COD and BOD_5 removal efficiencies by anaerobic pond are 46.0% and 90.0%,respectively (as shown in table1).But the color intensity of the molasses wastewater is not changed by above anaerobic pond. The color intensity (optical density at 475 nm) of the stillage and An-MWW were 84.4 units and 78.2 units, respectively.

Chemical removal efficiency of MWWs by various kinds of coagulants : Two kinds of wastewater as stillage and An-MWW were used for checking the efficiencies of coagulants. The $FeCl_3$, Alum and CaO were used as the coagulants. The results are shown in table 2. In the case of stillage, the pH of stillage was adjusted to 12, 12 and 3 before coagulation by $FeCl_3$, Alum and CaO, respectively. And the pH of supernatant of reaction mixtures was 2.0, 5.0 and 12.0 respectively The color and COD removal efficiencies of stillage were 90.52%, 89.57% and 94.31% and 74.11%, 77.59% and 76.62% when the coagulants were $FeCl_3$, Alum and CaO, respectively. In the case of An-MWW, the pH of An-MWW was adjusted to 10, 8 and 5 before coagulation by $FeCl_3$, Alum and CaO, respectively. The pH of the supernatant of the reaction mixtures was 2.0, 4.2 and 12, respectively. The color and COD removal efficiencies of An-MWW were 98.72%, 97.57% and 93.61% and 95.86%, 89.36% and 80.38% when the coagulants were $FeCl_3$, Alum and CaO, respectively.

Molecular weight distribution of MWWs: The results of molecular weight distribution of stillage and An-MWW solution by gel filtration chromatography ars shown in fig.1. The results show that the stillage contained the larger molecular weight of color substances while the An-MWW solution contained smaller molecular weight of color substances.

Effects of aeration on the chemical properties of MWWs: The stillage and An-MWW were aerated with and without sludge-added (2,000mg/L) for 96 hrs. The results are shown in table3. The oxygen did not show any effects on the decrease of the color intensity of the stillage and An-MWW after aeration for 96 hrs. The color intensity of stillage and An-MWW were the same as before being aerated. But in the case of stillage and An-MWW which were aerated with sludge, the color intensity of both MWWs decreased rapidly after being treated for 12 hrs. The color intensity of stillage and An-MWW were reduced by about 45.14% and 9.85%, respectively after being treated by aeration with sludge for 96 hrs. The molecular weight patterns of color substances in treated MWWs solutions are shown in fig.2 and fig.3. The results show that molecular weight patterns of the MWWs solution did not change after being aerated for 96 hrs. But the sludge which was added in the MWWs solution could adsorb color substances. The larger molecular weight of color substances could be easily adsorbed by sludge.

Effects on the pretreatment by aeration on color removal efficiencies in chemical coagulation step: The results are shown in table4. The stillage and An-MWW solutions were pretreated by aeration with and without sludge-added before coagulation by suitable chemical agents. The color substances of stillage after aeration with and without sludge-added for 96 hrs were coagulated by CaO in the concentration of 50 g/L and 60 g/L, respectively. And the An-MWW which was aerated with and without sludge-added for 96 hrs was coagulated by $FeCl_3$ in the concentration of 20 g/L and 12.5 g/L, respectively. The results are shown in table3. In the case of stillage, the color, COD and BOD_5 removal efficiencies by CaO were 93.36% and 95.25%, 72.79% and 63.54% and 67.11% and 69.32%, respectively when the stillage was aerated without and with sludge-added, respectively. In the case of An-MWW, the color, COD and BOD_5 removal efficiencies by $FeCl_3$ were 98.85% and 99.65%, 72.79% and 99.42% and 98.68% and 84.73% and 80.38% respectively when the stillage was aerated without and with sludge-added, respectively.

4. Discussion

The results of the chemical analysis of stillage of Sang som alcohol distillery factory showed that the impurities in the wastewaters were quite high. For example, the BOD_5 and COD were 30,000 mg/L and 110,000 mg/L. For the treatment system, so many researchers have tried to apply biological treatment processes, chemical treatment processes or both biological and chemical treatment processes for treating this wastewater. But up to now, they could not select a suitable treatment process for treating the waste water from the alcohol factories. The conventional biological treatment process commonly used in the alcohol factory such as anaerobic process and activated sludge process could not treat the wastewater to the standard requirement of the government[11]. In this factory, they used a big anaerobic pond (67,200 M^3 volume) for keeping and treating the MWW from the factory (HRT of the system was 220 days) The chemical properties such as BOD_5 and COD of the wastewater were reduced by about 90.0% and 46.0%, respectively. However, the impurities in the treated wastewater were still high. And also, the color substances still remained in the treated MWWs. It meant that the conventional biological wastewater treatment system could not remove the color substances from MWWs [1,2,3,4]. For the Sang som alcohol distillery factory, the degree of the color intensity could not be reduced by the microorganisms in the anaerobic ponds. The color intensity as optical density at 475 nm of the stillage and An-MWW were 84.4 units and 78.2 units, respectively. Sirianuntapiboon S. et al [6]reported that some microorganisms such as fungi and bacteria could adsorb the color substances from the molasses wastewater. But the adsorption ability of the microorganisms was effected by physical and chemical conditions such as the concentration of color substances, pH and toxic substances. For example, stillage had to be diluted about 10 times of the original before treating by above biological process. From the results in our experiments, the best coagulant which we used for removing the chemical impurities together with color substances from the stillage and AN-MWW were CaO and $FeCl_3$, respectively due to the low cost, highest color removal efficiency of color substances together with high chemical impurities removal efficiencies in MWWs and widely used in the chemical coagulation process [7,8]. In our previous paper [6,13,14] we found that some microorganisms could adsorb and change the molecular structure of color substances. Then, we try to use the biological treatment processes such as aeration with and without sludge-adding for pre-treating the MWWs before chemical coagulation step for increasing the removal efficiency in the chemical coagulation step. The results showed that the color substances of MWW could not be reduced by aeration. But the amount of CaO used in the coagulation step was reduced from 83g/L to 50 g/L. when the stillage was aerated with sludge-added for 96 hrs (table 3 and table 4). For the observation of molecular weight pattern of the aerated stillage compared with un-aerated stillage, we could not find any difference in the molecular weight patterns of both kinds of stillage. From the results above, we can say that aeration could not reduce or increase the molecular weight of the color substances. But aeration might change some functional group of the color substances to be suitable for coagulants to coagulate [12,13,14]. And the color removal efficiencies of raw stillage and aerated stillage with sludge-added were 94.31 % and 95.25 %, respectively (table 2 and 4).

The results on the pretreatment of the MWWs by aeration with sludge-added before coagulation of color substances gave interesting results as shown in table3 and table4. The color intensity of stillage and An-MWW after aeration with sludge-added was reduced by about 45.14% and 9.85%, respectively because the sludge (microorganisms) which was added in the stillage could adsorb some color substances onto the microbial cells [13,14,15]. For the determination of molecular weight patterns by gel filtration chromatography, we found that some parts of large molecular weight pigments were adsorbed by microorganisms (sludge). And the remaining color substances in the pretreated stillage were easily removed by suitable coagulants which were mentioned above.

For application, we believe that the above processes could be combined in the conventional wastewater treatment process which are normally used in the alcohol distillery factory. For example, stillage or An-MWW was pretreated by aeration with sludge-added for 96 hrs. After that, The remaining color substances and other impurities (BOD_5, COD and TKN) in the supernatants of pretreated MWWS could be easily removed by small amounts of coagulant (CaO). Then, the supernatants of treated MWW could be treated by conventional biological treatment process which are commonly used in the wastewater treatment plant of alcohol factories, such as aerated lagoon, oxidation pond for treating the remaining impurities. Then, the quality of treated wastewater, using our recommended treatment process, would be at the standard level of department of industrial works of Thailand.

Tablel: Chemical properties of molasses wastewater from the wastewater treatment plant of Sang som alcohol distillery factory.

The stillage and An-MWW from wastewater treatment plant of Sang son alcohol distillery factory were collected for determination of chemical properties as described in the text.

Chemical properties	Stillage	An-MWW	% removal
pH	4.8	8.2	-
BOD_5 (mg/L)	30,000	3,000	90.0
COD (mg/L)	100,000	54,000	46.0
TKN as N (mg/L)	1,200-1,900	33,000-39,000	-
Phosphorus as P (mg/L)	100-200	70	30-60
Total solid (mg/L)	110,000	45,000	59
Potassium (mg/L)	5,000-7,000	5,000-7,000	-
Reducing sugar(%)	0.2-2.5	0.4	80-84
Color intensity (OD_{475})	84.4	78.2	7.3

Table 2: Color and COD removal efficiencies of MWW by various kinds of coagulants (Alum, $FeCl_3$ and CaO).

The stillage and An-MWW from wastewater treatment plant of Sang som alcohol distillery factory were coagulated by three kinds of coagulants ($FeCl_3$, Alum and CaO) under optimal condition as described in the text.

Type of molasses waste water	Types of chemical agents and suitable concentration	Chemical properties							
		pH		color intensity (OD_{475nm})			COD (mg/L)		
		initial pH	pH of reaction mixture	Before	After	% Removal	Before	After	% Removal
Stillage	$FeCl_3$ 50 g/L	12	2.0	84.4	8.0	90.52	105,260	27,250	74.11
	Alum 33.5 g/L	12	5.0	84.4	8.8	89.57	105,260	23,590	77.59
	CaO 83g/L	3	12.0	84.4	4.8	94.31	105,260	29,870	76.62
An-MWW	$FeCl_3$ 29 g/L	10	2.0	78.2	1.0	98.72	39,936	1,654	95.86
	Alum 17g/L	8	4.2	78.2	1.9	97.57	39,936	4,251	89.36
	CaO 50 g/L	5	12.0	78.2	5.0	93.61	39,936	7,834	80.38

Table 3: Effects of aeration on the chemical properties of molasses wastewater.

The stillage and An-MWW were aerated with and without sludge-added for 96 hrs as described in the text.

Types of treatment	Types of molasses wastewater					
	Stillage			An-MWW		
	Color intensity			Color intensity		
	Before treated	After treated	% Removal	Before treated	After treated	% Removal
aerated for 96 hrs	84.4	84.4	0	78.2	78.2	0
aerated with sludge-added for 96 hrs	84.4	46.3	45.14	78.2	70.5	9.85

Table 4: Effects of pretreatment by biological process on color removal efficiencies of the molasses wastewater in the chemical coagulation step.

The MWWs were pretreated by aeration with and without sludge-added for 96 hrs. Then, the supernatants of pretreated MWWs were coagulated by suitable coagulants (CaO for stillage and $FeCl_3$ for An-MWW)at the optimal condition as described in the text.

Type of Molasses waste water	pre treatment	Chemical treatment	Color intensity ($OD_{475\ nm}$)			COD (mg/L)			BOD_5 (mg/L)			pH	
		type of chemical agent	before	After	% Removal	Before	After	% removal	Before	After	% removal	Before	After
Stillage	aerated without sludge	CaO 50 mg/L	84.4	5.6	93.36	80,500	21,908	72.79	15,200	5,000	67.11	5.0	12.2
	aerated with sludge	CaO 60 mg/L	46.3	2.2	95.25	27,760	10,120	63.54	11,250	3,452	69.32	6.0	12.4
An-MWW	aerated without sludge	$FeCl_3$ 20 mg/L	78.2	0.9	98.85	27,650	160	99.42	550	84	84.73	8.6	2.0
	aerated with sludge	$FeCl_3$ 12.5mg/L	70.5	0.25	99.65	2,280	30	98.68	260	51	80.38	8.6	5.4

<u>Remark:</u> The pH of stillage and An-MWW were adjusted to be 5 and 12, respectively before being coagulated by suitable coagu

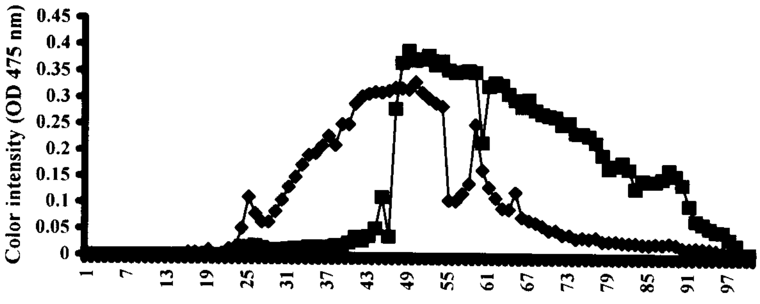

Fig.1: Comparison of molecular weights distribution of stillage and An-MWW on Sephagex G-50 column. Symbols: ◆ , stillage solution; ■ , An-MWW solution.

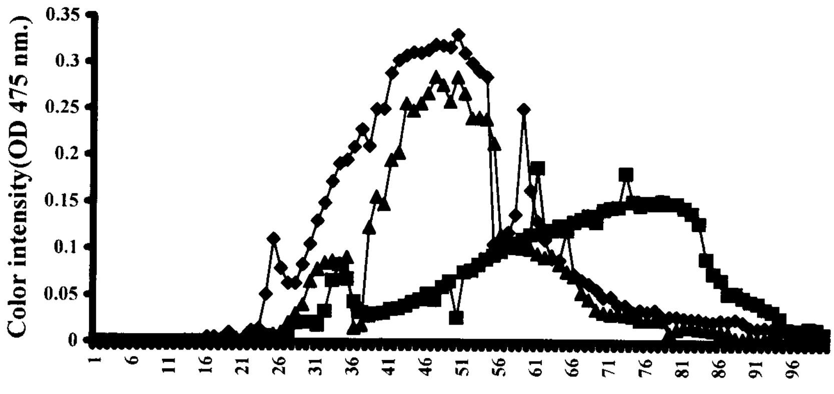

Fig.2: Gel filtration chromatograms of stillage before and after being treated by aeration with and without sludge-adding for 96 hrs on a Sephadex G-50 column. The molecular weight distribution of color substances in stillage before and after being treated by aeration with and without sludge-added (final concentration of the mixer being was 2,000 mg/L) were determined by gel filtration chromatography on a Sephadex G-50 column as described in the text. Symbols: ◆, stillage solution; ▲, aerated stillage; ■, the stillage after aerated with sludge.

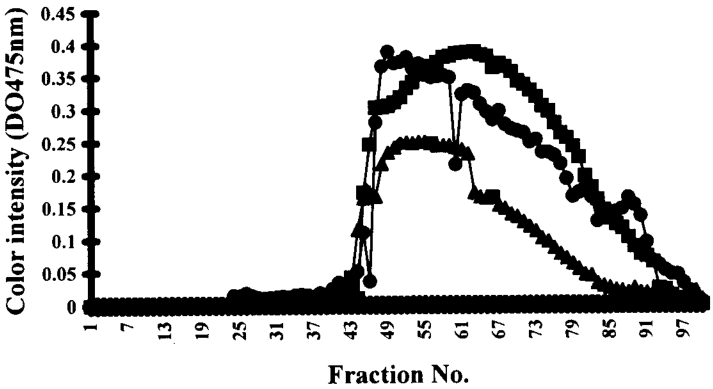

Fig.3: Gel filtration chromatograms of An-MWW before and after being treated by aeration with and without sludge-added for 96 hrs. The molecular weight distribution of color substances in An-MWW before and after being treated by aeration with and without sludge-added (final concentration of the mixer was 2,000mg/L) were determinated by gel filtration chromatography on a Sephadex G-50 column as described in the text. Symbols: ●, An-MWW solution; ■, aerated An-MWW; ▲, the An-MWW after aeration with sludge

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