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WASTEWATER QUALITY CONTROL ANALYSIS IN THE PHARMACEUTICAL INDUSTRY USING PROCESS CAPABILITY APPROACH

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ABSTRACT

Wastewater produced from the water management process must be in accordance with environmental quality standards in order to reduce the potential risk for environmental damage and human health. One of the pollutant compounds in wastewater that is the problem in this article is Chemical Oxygen Demand (COD) which is not in accordance with the environmental quality standards of the Indonesian government. The purpose of this study is to analyze the process capability of wastewater treatment, especially for COD pollutant parameters. Furthermore, several methods are used in this study such as IMR control maps, process capabilities, and Fishbone diagrams. The results show that the COD parameter has an average Cp index value of around 1.03 and an average Cpk of approximately 0.44. Therefore, it can be concluded that the wastewater management process still needs to be improved to produce wastewater that conforms to specified standards. This research can be the basis for subsequent research related to improving the quality of wastewater management processes.

Keywords: wastewater, COD, IMR, process capability, pharmaceutical industry.

INTRODUCTION

Waste discharged directly without treatment will potentially pollute the environment and reduce the quality of human health. The difference in the type of process and the dimension of the production has a significant correlation to the degree of difference in the variation of waste contamination in the industry. One type of waste produced in the production process is wastewater. Understanding wastewater is water that has been used by humans in various activities. The wastewater can come from household activities, offices, shops, public facilities, industry, and other places. Furthermore, wastewater is unused used water that is produced from various human activities in utilizing clean water. One industry that has the potential to produce wastewater is the pharmaceutical industry.

In general, wastewater is produced through washing equipment in the pharmaceutical industry. Even though the wastewater is discharged in small volumes, it has properties that are harmful to the environment due to a large number of organic pollutants. During the drug manufacturing process, various wastes and contaminants are generated such as organic matter, debris, dirt, sand, pollution, toxic materials, non-toxic, polymers. Wastewater classification is appraised related to temperature, pH, total suspended solids (TSS), total dissolved solids (TDS), biochemical oxygen demand (BOD), chemical oxygen demand (COD), oil and fat, chloride and sulfate for influents and effluents.

The development of chemical analysis technology advances over the past two decades shows the potential for the detection of chemicals in surface waters at low concentration levels (ng/L to µg/L). This is commonly known as micro pollution. Some micro pollutants can be found in agriculture, industry and various daily use products such as body care products, drugs and cleaning agents [1]; [2]. Moreover, the negative impact of micro pollution on aquatic ecosystems, the environment, and human health has been proven by several researchers [3]; [4]. Several of these products are produced from the pharmaceutical industry process.

Various activities in the pharmaceutical industry process have the potential to produce liquid waste which can be categorized as a toxic and hazardous material. For example, antibiotics, analgesics and anti-inflammatory drugs which are toxic contaminants. The study by Stackelberg *et al*, underline that the presence of hazardous waste has the potential to cause serious damage to surface water [5]. In addition, dynamic compounds such as liquid regulating drugs, analgesics, antibiotics, antiseptics, hormones, and chemotherapy are also detected in the flow of wastewater and groundwater sources. This compound has a different concentration level in the body of water that holds wastewater from the processing of liquid waste. Sim *et al.*, describe that there are concerns about the potential for hazardous compounds in drinking water that do not meet the specified clean water quality standards [6]. Thus, this may affect human health through chronic exposure.

Throughout the history of human life, the issue of science and wastewater treatment and reuse is relatively not a new issue. A study by Angelakis and Snyder observed the reuse of urban wastewater that did not go through the wastewater treatment process to divert wastewater out of urban settlements for centuries [7]. The application of domestic wastewater is an activity that is commonly carried out by humans and has undergone various stages of improvement and development. This encourages a better understanding related to process and processing technology and ultimately related to water

quality standards [8]. For example, research conducted by Sari *et al*. utilizes shrimp shell waste as bio-coagulation to purify urban wastewater [9].

In addition, an increase in environmental awareness related to industrial waste encourages industries to be able to formulate waste management and prevention strategies into industrial processes. Numerous contamination inhibition opportunities can be implemented with significant economic remunerations for the industry, as well as reducing environmental contamination [10]. Wastewater discharged into the environment certainly have a sustainable impact. Thus, controlling the quality of wastewater is certainly needed with the aim that liquid waste can be controlled and the impact on the surrounding environment can be minimized. Preliminary studies have obtained information that the concentration of COD parameters has not shown compliance with quality standards, where there are still points that are outside the specification limits and this shows that the quality standards for wastewater are not related to the COD parameters. High or low quality of water parameters in the wastewater treatment process greatly affects the quality of wastewater that will be released into the environment. Thus, an analysis is needed to determine whether the wastewater treatment process meets environmental quality standard specifications.

The wastewater treatment process in the treatment unit requires the determination of the process capability index. This will make it an easier evaluation process. Thus, if the capability of the process is appropriate, the wastewater produced will also be good and meet the specified standards. Meanwhile, if the process capability is at a critical point, then more careful control can be made, both the capability of the process and the declining quality of wastewater. Thus, the purpose of this article is to analyze the value of process capability resulting from wastewater treatment, especially the COD parameters.

PROCESS CAPABILITY

Process capability is an analysis of variability relative to product requirements or specifications and to assist the development of production in eliminating or reducing much of the variability that occurs. Process capability is a critical performance measure that shows the process is able to produce according to product specifications implemented by management based on customer needs and expectations [11].

There are two ways to think about this variability:

a) The variability that is inherent or natural at any given time is 'instantaneous' variability
b) Variability includes time

Among the main data uses of the process capability analysis are as follows:

a) Estimates how well the process will meet tolerance
b) Assist product developers or designers in selecting or changing processes
c) Assist in the formation of intervals for controlling intervals between sampling
d) Define performance requirements for new tools
e) Choose among competing sellers
f) Reducing variability in the production process

A process is stated to have a good ability if the spread of regular deviations in accordance with the distribution of the specified limits. Thus, if the ratio of the range specified with the control limit is greater than one. In other words, the following ratio must be greater than 1:

$$Cp = \frac{USL-LSL}{UCL-LCL} = \frac{USL-LSL}{6\sigma} \quad \ldots\ldots (1)$$

Where:
USL: Upper Specification Limit
LCL: Lower Specification Limits: Standard deviation of the process = R / d2

Cp value = 1, if the specified standard limit is the same as the natural variation limit of the process (control limit), in this case, the process is said to be incapable; it has the potential to only produce undamaged products if the process is centered on the specified target. About 0.27 percent, or 2700 parts per million, is damaged.

Cp value> 1 if the specified standard limit is greater than the control limit, in this case, the process is potentially capable and (maybe) produce products that meet or exceed customer requirements. Cp value <1 if the standard specified limit is smaller than the control limit. The process is said to be incapable.

Actual Process Capability, Cpk: The reason why Cp> 1 does not mean that the process does not produce defects, this is a range of control limits that may be smaller than the specified standard limit, but if the process is not centered on the specified target, then one side of the control limit might exceed the specified limit.

If the process is not centered on the specified target, Cp will not be too informative because it will only differentiate between the two ranges (the process control limit and the specified standard boundary), but it cannot inform whether the process produces defects or not. In this case, another capability index is used to determine the ability of the process to respond to customer needs and that is Cpk.

Cpk measures how many production processes that actually meet the standard specifications. K in Cpk is called the k factor; it measures the degree of process deviation from the specified target.

$$Cpk = (1-k)Cp \quad \ldots\ldots. (2)$$

$$k = \frac{[(USL+LSL)\ /\ 2-X]}{(USL-LSL)/2} \quad \ldots. (3)$$

In the analysis method for quality improvement, the process capability criteria are usually used for the Cp and Cpk values as follows:

a) Value Cp = Cpk, indicating that the process is in the middle of its specifications.
b) Cp value> 1.33, the process capability is very good.
c) Cp value <1.00, identifies that the process produces products that are not according to specifications and are not capable.
d) A negative Cpk value indicates the average process is outside the specification limits
e) Cpk value = 1.0 indicates a process variation that is within one of the specification limits.
f) Cpk value <1.0 indicates that the process of producing products that are not according to specifications.
g) Cpk value = 0 indicates the mean, Cpk value equal to 1 means the same as the specification limit.

METHODS

The source of data that will be used as material for analysis in this study is the quality of water produced from the wastewater treatment process for the COD parameters. Data is taken from one of the pharmaceutical industry. Data on wastewater quality is obtained from the department responsible for wastewater treatment. The steps of analyzing the data of this study are as follows:

a) Conduct normality test data on the number of defects in wastewater for COD parameters.
b) Make an IMR control map based on the number of defects in wastewater for COD parameters.
c) Make Ishikawa diagrams of the most types of defect categories in wastewater for COD parameters.
d) Calculate the process capability of the wastewater treatment process for COD parameters.

RESULTS AND DISCUSSIONS

Based on the data collected from the COD parameter data, information is obtained as shown in Table-1 below. A normality test is carried out on the COD parameters of wastewater with a total of 30 data from each of the parameters that have been previously measured. Testing is done by the Kolmogorov-Smirnov method. Manually testing the normality of data in this study conducted with the following stages first the results of data collection are calculated first the average and standard deviation s, then sorted from the smallest data.

Table-1. COD Parameter Measurement Data.

No.	COD value	No.	COD value	No.	COD value	No.	COD value
1	**50**	**11**	**35**	**21**	**24**	**31**	**21**
2	38	12	27	22	42	32	18
3	46	13	26	23	36	33	23
4	47	14	25	24	25	34	23
5	31	15	22	25	17	35	38
6	33	16	28	26	27	36	37
7	41	17	41	27	24	37	46
8	39	18	32	28	35	38	34
9	37	19	21	29	18	39	37
10	28	20	34	30	16	40	33

Based on Table-1, then conducts the normality test, thus the results can be recapped in Table-2 below:

Table-2. Summary of Data Normality Test Outcomes.

Parameters	P-Value	Interpretation
COD	0.150	Normal

After the data is calculated the present value is then made a probability plotting graph with the results of the COD test results as the X-axis and percentiles as the Y-axis (Figure-1) and from the results compared with normal probabilities using Minitab 16.1.1 software using the Kolmogorov-Smirnov method. Based on this the data normality test is then performed, so the results can be summarized as Figure-1 below:

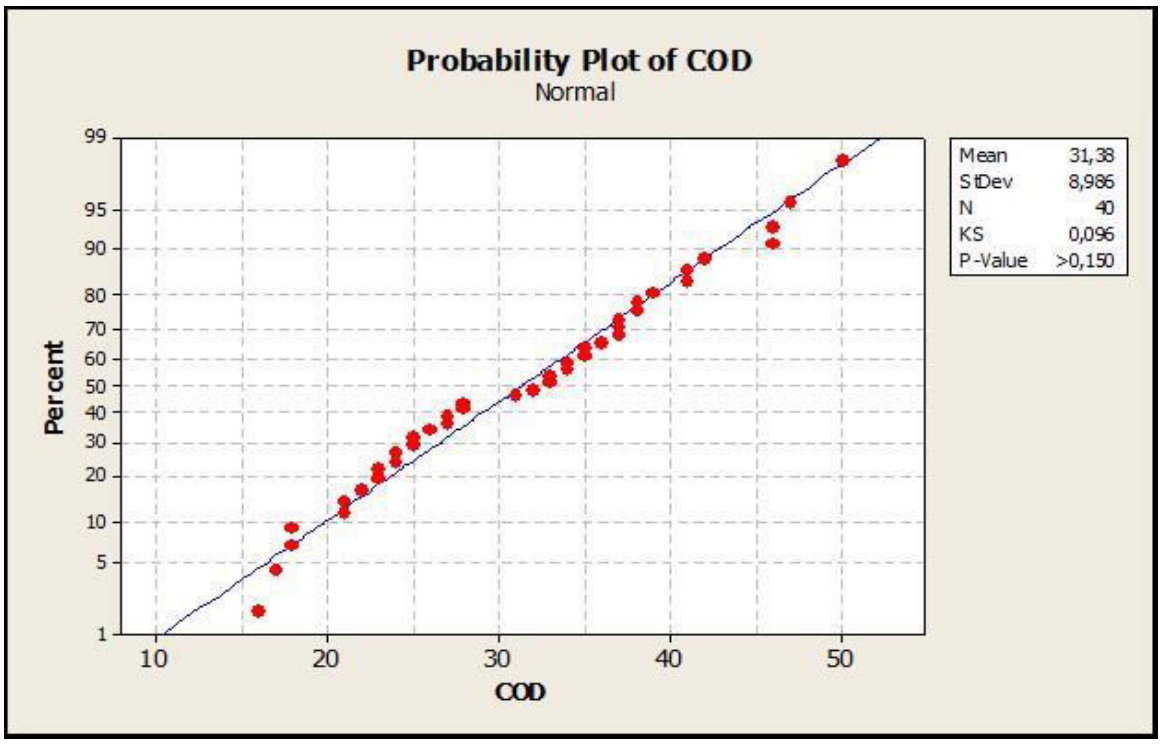

Figure-1. Normal Probability Plotting.

The I-MR control chart was chosen in this data processing because the wastewater treatment process is carried out continuously and the laboratory testing process is quite expensive so the sample testing data obtained are individual. This control chart serves to determine whether the performance of the wastewater process is within the control limits (in control), where the process runs well and is stable without any special cause influences.

$\delta = (MR)^{-} / d2 = 7.30769 / 1.128 = 6.478$ (the value of d2 is taken from the control chart constant table where for n = 2 the value is 1.128)

Next is calculating the central line (CL), upper control limit (USL) and lower control limit (LSL) with the following formula:

$CL = X^{-} = 31.375$
$USL = CL + 3\delta = 31.375 + 3\ (6.478) = 50.809$
$LSL = CL + 3\delta = 31.375 - 3\ (6.478) = 11.941$

Whereas for the moving range (MR) map is as follows:

$CL\ MR = (MR)^{-} = 7.30769$

$USL = D4\ ((MR)^{-}) = 3,267\ (7, 30769) = 23,874$ (D4 value is taken from the control chart constant table, where for n = 2 the value is 3.267)
$LSL = D3\ ((MR)^{-}) = 0$ (D3 value of the control chart constant table for n = 2 is 0)

Picture-2 provides the following information, there are three horizontal lines parallel to the central line, the lower control limit (LSL) and the upper control limit (USL). Flat axis states the number of samples studied, starting from the first sample, second and so on. The upright axis states the characteristics understudies such as average, percentage or others. The points are connected by lines, expressing the characteristics for each sample. If these points are in the area bounded by USL and LSL then the process is said to be in control. This means that the process takes place or operates with a reasonable cause as expected or runs due to the cause of a fixed system that is probabilistic. From the calculation results, as shown in Figure-2 above, the values obtained for the central line, upper control limit and lower control limit for both X and MR control charts are the same as manually calculating values and all data are within control limits. However, there are some data that is close to the control limit.

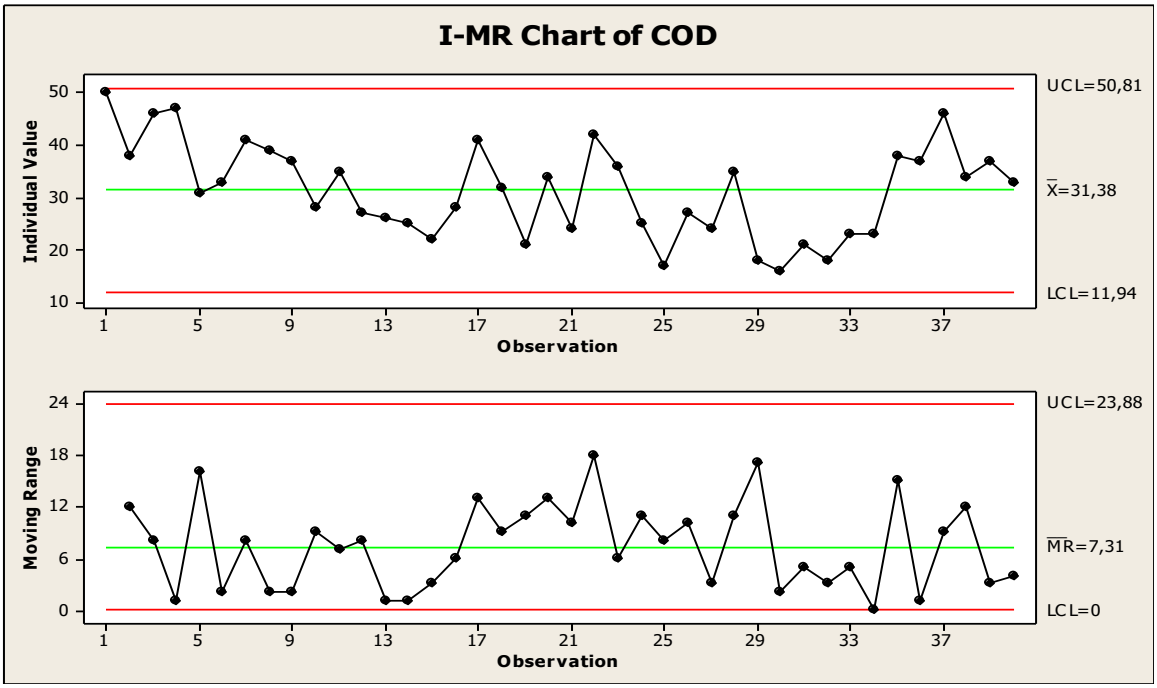

Figure-2. Control chart of I-MR COD.

Through testing or monitoring of the output quality of water treatment facilities, in this case, water quality with control chart tools and analysis of process capability will provide an overview for companies to see the performance of the water treatment process so far. In addition, the results of this analysis can also be input for companies whether the existing process needs to be improved.

The specifications set by the company for the lower specification limit (LSL) and upper specification limit (USL) are 0 and 40, thus the value of the process capability ratio (Cp) and the process capability index (Cpk) can be calculated as follows:

S = R / d2 = 7.30769 / 1.128 = 6.478 (d2 value is taken from the control chart constant table where for n = 2 the value is 1.128)
Cp = (USL-LSL) / 6S = (40 - 0) / (6 x 6.478) = 1, 03
CPU = (USL-X) / 3S = (40 - 31.375) / (3 x 6.478) = 0.44
CPL = (X-LSL) / 3S = (31.375 - 0) / (3 x 6.478) = 1.61
Cpk = min (CPL; CPU) = min (0.44; 1.61) = 0.44

While the possibility of water quality with COD values that are outside the specifications (out of specification) can be calculated as follows:

Za = (USL - X) / (S) = (40 - 31.375) / 6.478 = 1.33
Zb = (LSL - X) / (S) = (0 - 31.375) / 6.478 = 4.84
P = (1.33 <Z <4.84)

The possibility of a defective product below the specification is P (Z <1.33), in the standard normal distribution table, stated 0.091759 X 1000000 = 91759 ppm, while the possibility of defective product above the specification is P (Z> 4.84), in the table stated 1 - P (Z <4.84) = 0 (rounding), then the product above the upper limit is 0 X 1000000 = 0 ppm. Thus the total possibility of products outside the specifications is 91759 + 0 = 91759 ppm.

From the results of the analysis, a number that is relatively similar to the manual calculation is produced, namely a Cp value of 1.03 and a Cpk value of 0.44 and a probability of out of specification 91539.74 Ppm.

Research conducted by Rimantho *et al*., Shows that the raw water treatment process of ALT and pH parameters in the pharmaceutical industry also has small Cpk values of around 0.58 and 0.67 respectively [12].

Based on the calculation of process capability, the sigma value can be seen as the Table-3.

From Table-3 above regarding the stages of sigma value determination, the company has a maximum company specification limit of 40 as an alert zone before reaching the critical limit set by the government of 80, it can be seen that the sigma value for the current process is 2.83. Seeing this, it is necessary to find the root cause of the occurrence of conditions above the alert zone that can be done to prevent and prevent failure in the water treatment process.

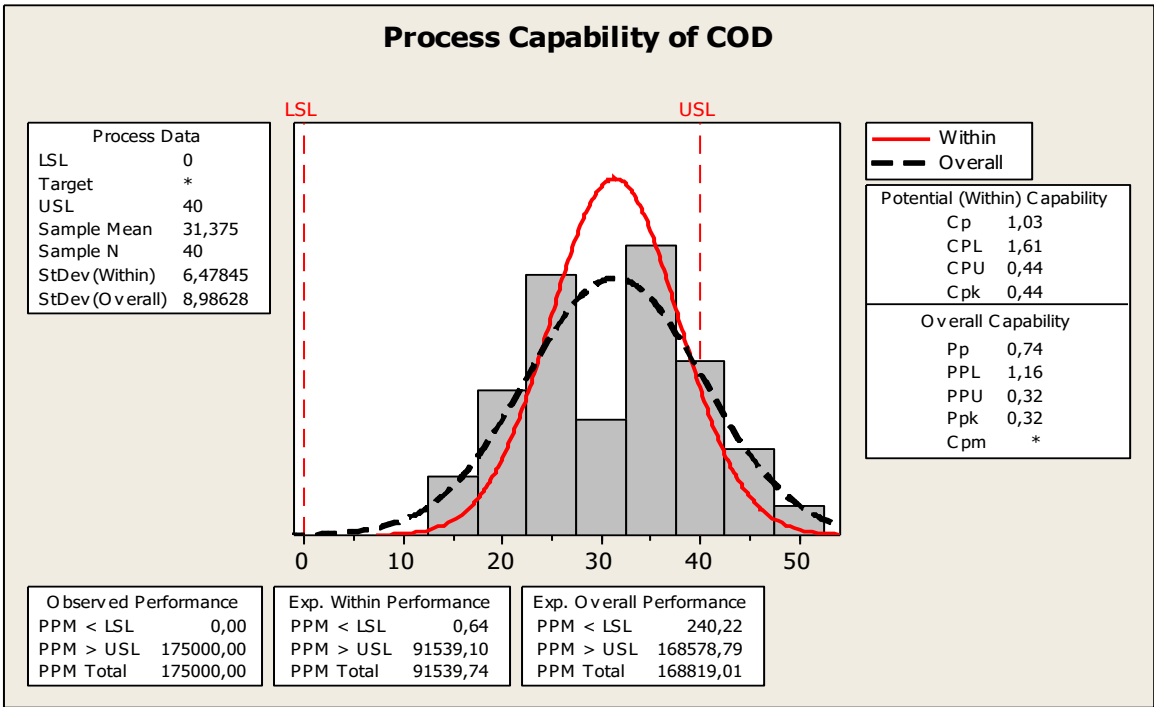

Figure-3. Analysis of Process Capability.

Table-3. Stages of Determining the Sigma Value of the Waste Treatment Process.

Steps	Action	Formulation	Result
1	What process do you want to know	-	Wastewater treatment process
2	Set the upper specification limit	USL	40
3	Set the lower specification limit	LSL	0
4	Determine the target specification value	T	20
5	What is the mean value of the process	X-bar	31,375
6	What is the standard deviation of the process	S	6,478
7	Calculate the probability of failure that is above the USL value per one million chance (DPMO)	P[z ≥ (USL - X-bar) / S] x 1.000.000 *)	91759
8	Calculate the probability of failure below the MSM per one million chance (DPMO)	P[z ≤ (LSL - X-bar) / S] x 1.000.000 **)	0,793
9	Calculate the probability of defects per one million opportunities (DPMO) produced	(Step 7) + (Step 8)	91759,793
10	Convert DPMO (Step 9) into sigma value	-	2.83
11	Calculate the capability of the above process in a Sigma value measure	-	Sigma value is 2.83

*) P [z ≥ (USL - X-bar) / S] x 1.000.000 = P [z ≥ (40 - 31,375) / 6,478] x 1.000.000 = P [z ≥ 1,33] x 1.000.000 = [1 - P (z ≤ 1, 33)] x 1.000.000 = [1 - 0.908241] x 1.000.000 = 91759

**) P [z ≤ (LSL - X-bar) / S] x 1.000.000 = P [z ≤ (0 - 31.375) / 6.478] x 1.000.000 = P [z ≤ - 4, 84] x 1.000.000 = 0,000000793 x 1.000.000 = 0,793

After the process capability index and sigma value as a quality baseline have been successfully established, the next step is to identify the factors that cause quality problems. Causes that affect the quality of wastewater produced by established company quality standards. The determination of these factors is guided by the concept of 4M plus 1E (Man, Machine, Material,

Method, and Environment). Basically, fishbone diagrams are used to present the cause of the problem graphically with the aim of identifying the cause of a problem, finding the causes and taking corrective action, helping to investigate further factors and selecting the methods used to solve the problem.

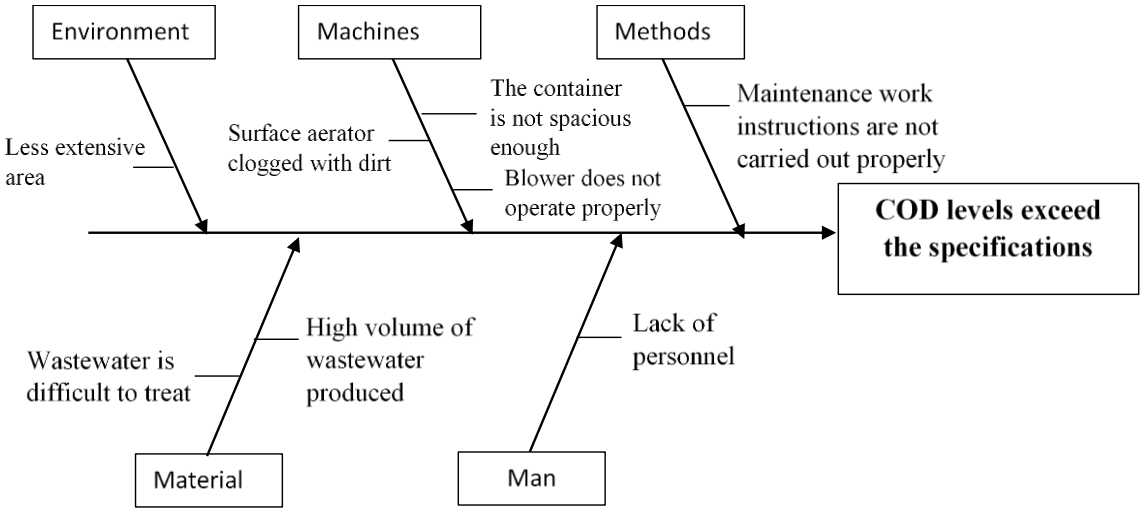

Figure-4. Wastewater Treatment Fishbone Diagram of COD parameters.

From the results of brainstorming activities, fishbone diagrams are then made to clearly know what problems are causing mismatches in the water quality, the results are presented in Figure-4. Furthermore, the proposed improvement is given based on the problems faced by the decision-makers in the company related to the ability of the water treatment plant installation process that is not good as specified by the company with the aim of improving water quality as follows:

a) Propose an experiment to reduce the concentration of production waste in the laboratory scale in advance to find out what materials can optimally reduce the content of production waste.
b) Propose that the giving of yeast when the condition of visually activated sludge has begun to black.
c) Propose an experiment to add yeast in a lab-scale beforehand to find out the optimal concentration in adding yeast.

CONCLUSIONS

The conclusions obtained from this study indicate that the wastewater treatment process has not been controlled statistically. This can be shown from the value of the process capability index (Cpk) which is only 0.44. This gives an indication that the wastewater treatment plant does not have good process capability with the possibility of the system producing a quality of liquid waste that is out of specification 91759 ppm. Factors that cause deviations in the quality of critical parameters that occur from water quality are caused by high volumes of production waste, production matrices that are difficult to process and microbes in saturated sewage sludge making it vulnerable to death. Improvements that can be made to these conditions is to make the initial waste treatment process (pre-treatment) such as the chemical coagulation process. This can be done by experimenting in a lab-scale to find out the optimal material and quantity in addition. Then conduct experiments on a laboratory scale to find out the addition of yeast with optimal concentrations so that microbes in activated sludge can stay alive.

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