

An Electroanalytical Instrument Equipped with Wireless Communication Network and Graphical User Interface for Real-time Monitoring Wastewater Status from Batik Industry

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ABSTRACT

Effluent discharge from the Batik Industries need to adapt the continuous monitoring system in order to cope with the growing economic. Non-invasive analytical instrument was designed in this work for real-time monitoring of wastewater status from batik factories. Compared with existing instrument, this instrument was simplified with wireless and portable, maximal convenient operator with aided Graphical User Interface, and can be applied for field work such as installing sensor at final discharge point. In this paper, a wireless instrument for determine conductivity, temperature and pH as an indicator for wastewater status was demonstrated and characterized. The synthetic solution was prepared for quality data references, data known and apply correction, robustness and interferences effect. The development of algorithm and mathematical formulation was established in order to obtain the directly measurement method for data acquisition and transmission. To demonstrate the performance of the instrument, the data comparison was made with the Inductive Couple Plasma (ICP-OES) for laboratory control sample. Finally, form the data analysis confirmed this instrument could provide wastewater status in Batik's Effluent in a convenient and non-invasive manner, and simultaneously enhance the awareness of batik entrepreneurs regarding to environmental pollution issues.

Keywords: *Monitoring, Wireless, Graphical User Interface, ICP-OES.*

Introduction

Introduction on industrial wastewater

Industrial pollution is one of the largest contributors to the global pollution whereas the waste produced should be control by implementation and enforcement the law of regulation [1]. The waste from by-product manufacturing distributing harmful pollutant into water, air and soil, risking of human health and environment sustainability [2]. Water pollution is one of the serious ecological problem due to consequently threat human and aquatic life [3].

The major source of water pollution is industrial wastewater discharge [4][5]. Industrial wastewater are classified into different types including organic content and inorganic content [6]. Organic content do not dissolved in water and has covalent bond such as phenol, formaldehyde, nitrobenzene and PCB. Inorganic content can be dissolved in water and has ionic or electrovalent bond such as pH, salt, heavy metals and Sulphur. The composition of water in wastewater is 99.93% and 0.07% is total solid which is summation of total dissolved solid and total suspended solid.

Current practice from batik industry in Malaysia

In Malaysia, batik industry has rapidly growing industry with an annual around RM 160 million according The Star Online, 25th September 2014 [7]. However, advancement in environmental and waste management is imperceptible in batik factories [8], negatively it has the potential impact to the environment [9]. The Kelantan Department of Environment (DOE) reported that factories has lowest level of compliance with the regulation which is 65% (DOE Kelantan Report, 2011).

According to the literature survey, wastewater discharge from most batik factories is released directly into environment without any prior treatment process [10]. In addition, conventional treatment process do not promises to remove chemical and specifically dyes and fixing reagents to meet the department's law with stringent regulations [11]. The production of batik utilize a lot of chemicals including synthetic and azo dyes which contain highly concentration of pollutants such as carcinogenic, colour pigment and toxic heavy metal, hence it cause pollution in aquatic habitat and environment [12][13]. Recently, many researcher starts to study the wastewater from batik factories that contains metal toxicity due to pollution problem endangering human health. Therefore, this paper introduce instrument to enhance the awareness of wastewater status from batik factories [14].

Environmental threat on metal toxicity

Heavy metal is the metal in aqueous state that can give detrimental health effect to human even at low concentration [15]. It cannot be degraded then consumed by human through drinking and entering the food chain [16][17]. Many exposure of heavy metal to human can effect organ and nervous damage, cancer, reduced growth, and extremely death [18][19].

Table 1: Heavy metal threat on human health

Heavy metal	Health Effect
Zinc (Zn)	Lethargy, neurological sign, depression, damage to nervous system and thirst [20]
Nickel (Ni)	Human carcinogen, dermatitis, nausea, coughing and chronic asthma [21]
Copper (Cu)	Insomnia, Wilson disease, liver damage, stomach and intestinal irritation [22]
Manganese (Mn)	Inhalation or contact causes damage to central nervous system [23]
Lead (Pb)	Fetal brain damage, congenital paralysis, sensor neural deafness, circulatory system, nervous system and kidney diseases [24]
Cadmium (Cd)	Human carcinogen, renal dysfunction, kidney damage, lung disease, bronchitis, gastrointestinal disorder, bone marrow, increase blood pressure and cancer [24]

Determination of heavy metal ions

For establish equipment nowadays has a great standards to determine heavy metal ions in laboratory [25]. The most dominant techniques are Inductive Coupled Plasma Mass Spectrometry (ICP-MS), Inductive Coupled Plasma with Atomic Emission Spectrometry (ICP-AES) [26][27], Atomic Absorption Spectrometry (AAS), Atomic Emission Spectrometry (AES), and Cold Vapor Atomic Absorption Spectrometer (CVAAS) [28][29]. These modern methods use optical detection are more selective and sensitive compare to conventional method [30], however there are not convenient for fields work such as batik factories because it require specifically techniques, long time storage, high cost and bulky. Bioanalytical techniques such as nuclei acid through DNA probes also used to trace heavy metal ions [31]. However, this development techniques recently useful for food and clinical application [32]. In general, most literature emphasizes electroanalytical technique for field

work instrument development, real-time and online wastewater status due to high performances such as low detection limit, good stability and wide linear response [33][34].

Electroanalytical technique for determine wastewater status using conductance measurement

Electroanalytical is one of the method to analyses heavy metal ion in analytic solution based on measurement of potential (volt) and current (ampere) in interfacial process. This technique can be classify according to their aspects on the cell controlled and measured such as potentiometry, coulometry and voltammetry. Conductance is a classical analytical techniques that finds in more widely application due to relatively short time, easy of interpretation, maximal convenient operator and on-line measurement [35].

For recent application, conductance is used for measure the salinity and total dissolved ion, assess solvent purity, determine relative ion strength including functioning as ion chromatography, monitor dissolution kinetics, determine micelle concentrations, follow the course of some enzymatic reactions and provide basic thermodynamic data for electrolyte solutions [36][37]. This researcher use conductance measurement to study the corrosion process of copper in nitric acid HNO₃ [38], study the nanofluids that contains metallic and ceramic particles [39], monitor microbial growth and metabolic activity in food industry, and measure the contribution of various component of milk since the distribution of salt fraction in soluble and colloidal correlate to conductance value [40]. C. Charnier use combination of electrical conductivity value and pH in titrimetric method to determine ammonia nitrogen, volatile fatty acids and inorganic carbon concentrations [41].

It is realized that conductance measurement can be extensively provided more information instead of the ions determination, thus this paper study the wastewater status by using indication of conductivity, temperature and pH value.

Methodology

Sample preparation

All chemical reagent used in this experimental work are grade quality and the solutions was prepared in the deionized water. Heavy metal concentration was obtained from copper ICP standard traceable to SRM from NIST Cu (NO₃)₂ in HNO₃, 2-3% 1000mg/L Certipur, E-Merck. For preparation of 100mg/L copper stock solution, 10 mg copper standard was dissolved in 100mL volumetric flask. Standard working solution of 0-50mg/L with increment 5mg/L was prepared by appropriate dilution of the stock solution. Synthetic solution was prepared with various level of pH value whereas pH solution is obtained from buffer concentrate for 500 ml buffer solution, pH 5-

8 0.02 (20 GRAD C) Titrisol, E-Merck. Dye solution preparation using methylene blue with molecular formula $C_{16}H_{18}C_1N_3S$. Stock solution was prepared with an accurate weighed of methylene blue was dissolved in distilled water for 100mg/L. Standard solution, a series of simulated wastewater with mixture of known composition of copper, pH level, and dye concentration was prepared for extensively analysis. Concentration was determined by using inductive coupled plasma (ICP-OES).

Instrumentation

Figure 1 represents the measurement architecture system. The system includes a set of submersible water pumps, piping network system, sensing unit (electrical conductivity, pH and temperature) from dfrobot, RF95W LoRa wireless module, controller unit (Arduino mega 2560 and Arduino nano), amplifier circuit unit, power supply SPP1272 battery 12V 7.2AH, electronic GPI flow rate digital meter and hydraulic bench.

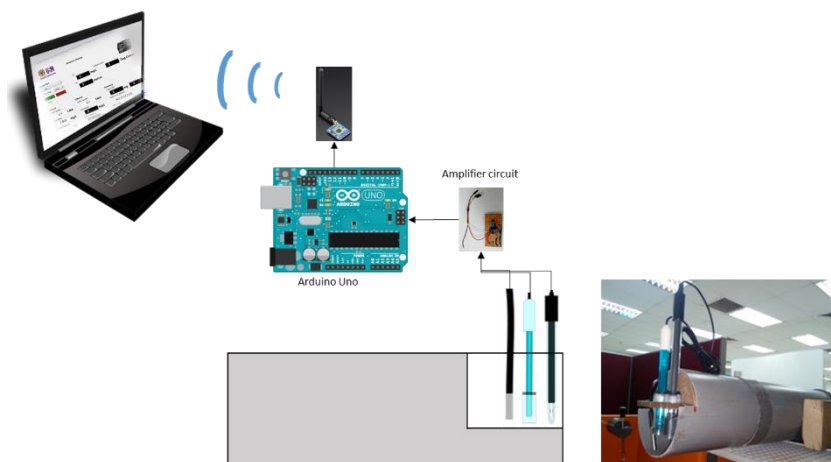


Figure 1: Measurement architecture system

Overall framework

The block diagram are shown in figure 2. It mainly includes five parts: sensing unit (input from physical phenomenon), signal conditioning (alter the voltage input), acquisition hardware (intermediate process of data analysis), transmission network (data transmit as input to monitoring terminal), and monitoring terminal (status display screen). Long range

antenna is incorporate with RFM95W using a lower license-free ISM frequency bands 433MHz. This radio has envelope area for field sensor network node around 2km line of sight. By considering low rate data transfer or small packet of data, this module is compatible for wireless instrument. From the data sheet, the data rate is around 19.2 Kbps (kilobits per second) since the data rate for this Arduino to the computer is 11.52 Kbps. The data will be store on the computer by initiating directory system. An electroanalytical system for sensing unit containing conductivity sensor, pH sensor, and temperature sensor is located on a final discharge point (FDP).

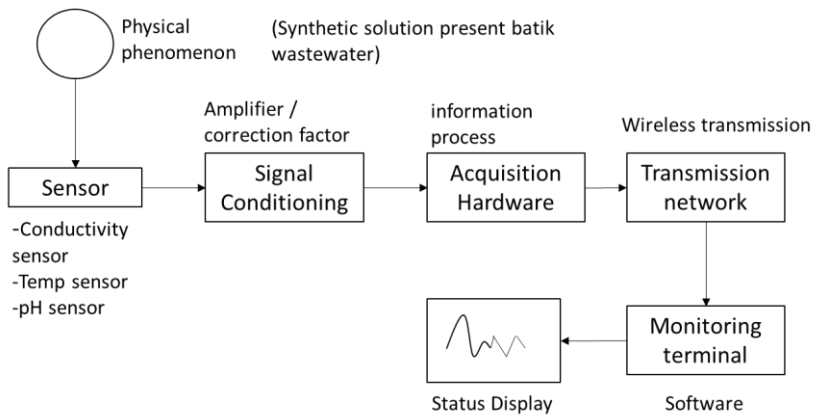


Figure 2: Overall framework

Data transmission

Figure 3 depict an illustration of real-time monitoring, the detail frame for network architecture is divided to two layers namely perception and application layer. The determination results are converted into electronic signal from sensing unit, hence it will be analysed and processed on application layer. The mathematical formulation and algorithm is established on controller programme. The final results will transfer as a small packet of data by using RF95W communication module to the monitoring terminal. In a perception layer, the small packet data will be received whereas the envelope area of transformation is capable in long distance. The mass data will be stored and used in visual C# to generate graphical user interface and display the wastewater status on monitoring terminal. Thus, the entrepreneurs of batik industry can monitor wastewater status regularly in real-time with convenient instrument and effectively.

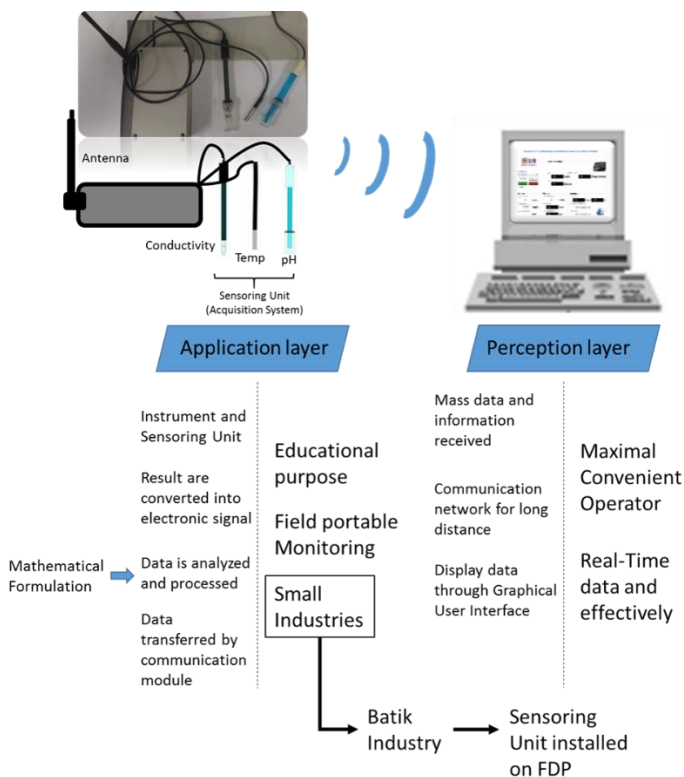


Figure 3: A schematic of wireless communication system

Graphical User Interface

In perception layer, Graphical user interface (GUI) that allow the operator or user interact with the instrument device, display an indication of wastewater status by graphical icons and visual indicators. This aided designed can be classify as significant part of software application programming which enhance the efficiency and maximal convenient operator for instrument. The detail window of the GUI is shown in figure 4 containing four parts (serial port setting, standard display result, influent/effluent solution reading, and wastewater status). An interface is developed in Microsoft Visual Software, a stored programme is connected with Arduino Uno board through serial port with baud rate 11.52 kbps. Directly measurement converted from voltage signal is display on standard display result. Influent / effluent solution reading resulted from metal concentration formulation determine the

capability of the treatment system. At last, wastewater status display fixation rate for the batik making process and quality of wastewater discharged.

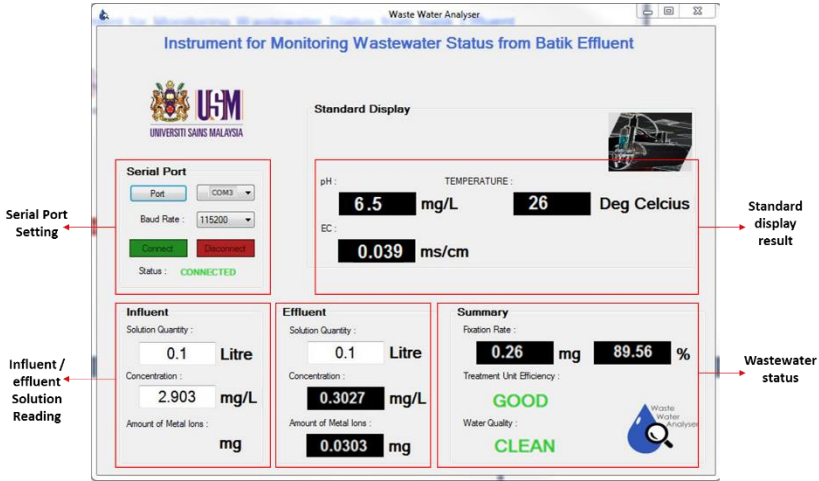


Figure 4: Graphical User Interface (display window on terminal monitor)

Robustness analysis

Temperature

The measurement of conductivity changes with variation temperature as thermal energy increase, Brownian motion will be increase [42]. The stability of temperature in this experiment is significant because it can affect the accuracy of measurement. The temperature coefficient, α can be expressed as;

$$\alpha = (d\chi/dt)/\chi \quad (1)$$

Where α is a temperature coefficient, t is a temperature, χ_i is a conductivity values that measure, and χ_{25} is conductivity measurement at temperature of 25 degree. Thus,

$$\chi_i = \chi_{25} [1 + \alpha(t - 25)] \quad (2)$$

The monitoring result is fluctuated around the reference value and this can be relate to environment factor such as temperature. In order to enhance the accuracy of the measurement, the temperature considered in mathematical formulation. As figure 5, AV is the average voltage from sensor input and

CV is the coefficient voltage where the temperature is consider to alter the voltage signal. The graph depict an accuracy for this instrument with references value, accuracy for AV is 97.94%, after considering temperature, accuracy for CV increase to 99.95%. However, this experiment was conducted in laboratory condition, thus the temperature difference is slightly low.

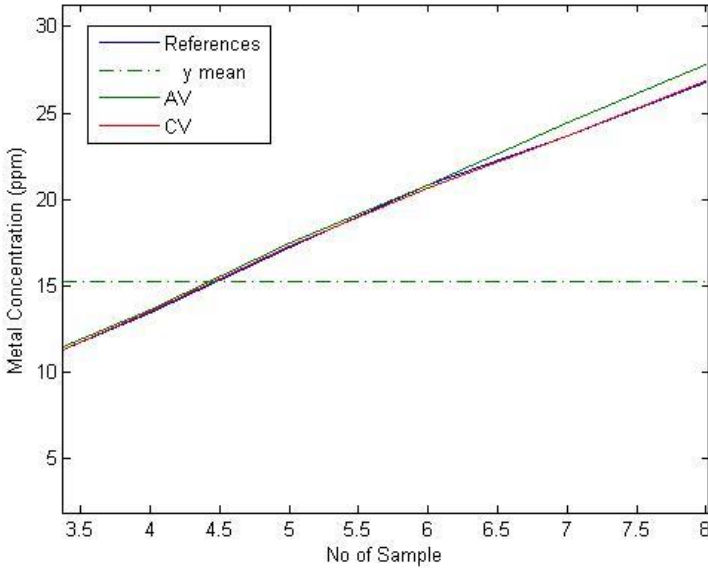


Figure 5: Influence of temperature consideration on accuracy of conductance measurement

pH

By concerning the effect of pH in measurement, this can be relate to the principal of electrostatic interactions. These interaction are between the charged surface of copper and the hydrogen ions H⁺ of the synthetic solution. At first, as mention in previous, temperature is a significant parameter that forces contributing to the stability of the readings. A voltage input from the pH sensor not prefer to convert directly into pH value. Mathematical formula is established to consider temperature in calculation as below;

$$pH Val = 7 + \frac{(V \text{ at } pH \ 7 - V \text{ input})}{sens + (temp \ sensor - temp \ at \ pH \ 7) \times 1.984 \times 10^{-4}} \quad (3)$$

These pH determination is consider necessary part to observe the interaction and the attitude if hydrogen ions in synthetic solution. Thus, figure 6 illustrates the hydrogen ion contents in solution based on pH measurement. At pH 7, the concentration is consider neutral. Increase one unit value of pH, the amount of hydrogen ions increase ten times. The mathematical formulation is established as;

$$pH = -\log[H^+] \quad (4)$$

$$[H^+] \text{ mg/L} = 10000e^{-2.303pH} \quad (5)$$

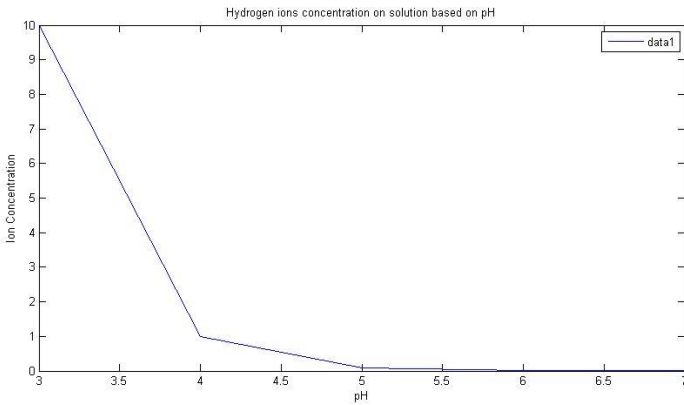


Figure 6: Hydrogen ions concentration on synthetic solution based on unit in pH

Flow rate

Figure 7 shows that flow rate of the solution is not effected the measurement readings. The flow rate will affect the concentration of metals ions dynamically as a physical disturbance (pH and DO) [43][44]. However, the response time for measurement to achieve stability slightly different as different flow rate. For static samples, response time $\tau = 26$ sec. At flow rate < 0.3 L/s, response time reduce slightly until flow rate > 0.4 L/s, response time maintained to $\tau \cong 20$ sec. This value determination is significant to understand the behavior of measurement in solution at initial value.

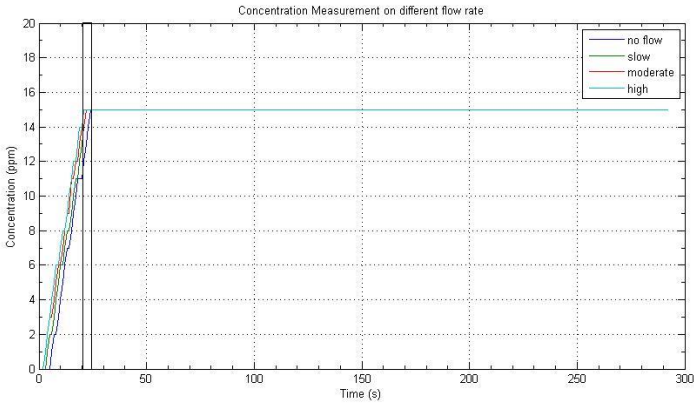


Figure 7: Response time, time required for sensor achieve uniform measurement

Calibration plots

The result for calibration plots is shown in figure. The instrument is calibrated by applying a standard references known solution as physical input and observe the system response. It is clearly shows that the coefficient of voltage (CV) signal proportionally to the concentration of metal, χ . The correlation by using least square method ($R^2 = 0.9999$) can be established as concentration, χ (mg/L) = $0.0482CV + 1.12$. When the trend line is set to y-intercept, $x = 0$, the quadratic equation is expressed as $\chi = 0.511CV$ at $R^2 = 0.9951$.

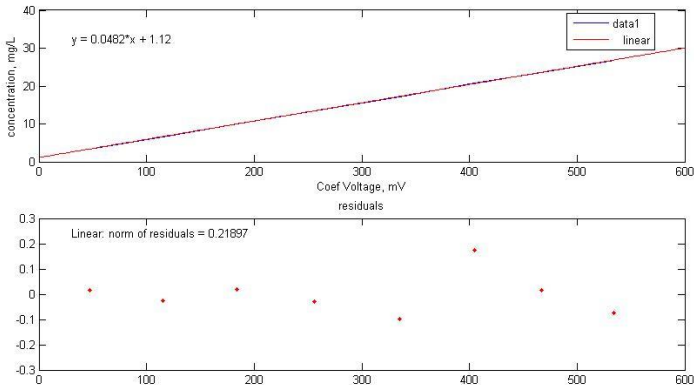


Figure 8: The relationship between the physical measurement variable (concentration) and the signal variable (coefficient voltage)

Instrument performance

As can be seen from table 2, an initial demonstration was performed using laboratory control sample to characterize performance of the instrument. To determine instrument detection limits, using $IDL = t \times \delta$, perform all calculations and result represent as 0.05 mg/L with accuracy 98.2% and accuracy is increase to 99.95% at 1.1 mg/L. The precision or reproducibility of this instrument by the standard deviation is 0.05087 mg/L. RPD for 20 samples not exceeded than 2% where the condition under control limit, $RPD = 0.3\%$.

Table 2: Instrument performance

Standard deviation, δ	Instrument Detection Limit, IDL	Sensitivity	Relative percent difference, RPD
$\sqrt{\frac{\sum_{i=1}^{274}(X_i - 1.860128)^2}{274 - 1}}$ SD= 68.95% 0.05087	IDL = 1.1 with accuracy 99.95%	0.001	$\frac{ D_1 - D_2 }{(D_1 + D_2)/2}$
	IDL = 0.05 with accuracy 98.20%		0.3%

In order to verify the mathematical formulation establish, a comparison of calculation from literature survey method is presented. Previously, a researcher evaluate total concentration of summation 15 elements of ions and its rms error. In their experimental, the total concentration of metal ions, C is plotted with electrical conductivity, χ values and adequacy of empirical equation result as linear dependence following equation that obtained least squares procedures ($R^2 = 0.997$) [42][45];

$$\chi = 25.104C + 909.85 \tag{6}$$

From Lenntech, they established calculator for simple calculation metal concentration by relation of electrical conductivity (EC) and total dissolved solid (TDS). This simple mathematical formulation is $TDS = 0.5 \times EC$. Lenntech also state that at high TDS value, the relationship changes to $TDS = 0.9 \times EC$. However, a researcher proved that concept is not to be true due to their experimental work. One of the reason is, the values for EC and TDS is equal during their experimental work. In addition, they state that conductivity is a function of dissolve substance and depends on valences of difference material compounds that present in electrolytic solution. The mathematical model established due to their valent metal compound which is mono-valent compounds ($EC = 0.052672+1.5025TDS$), di-valent metal

compounds ($EC = 1.072+1.31TDS$) and Tri-valent metal compounds ($EC = 0.0251879+1.49308$) [46][47]. Figure 9 illustrates comparison method from literature survey and this experimental work.

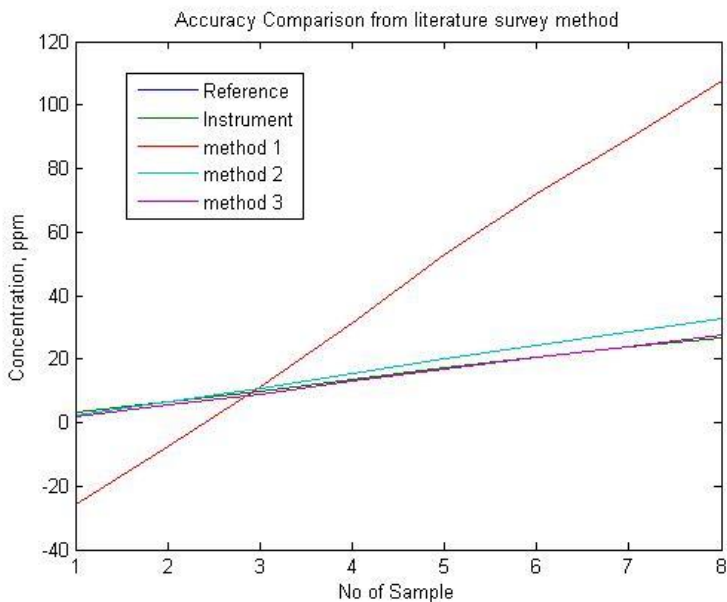


Figure 9: Accuracy comparison using mathematical formula of conductance measurement (a) instrument ; this research 99.95%, (b) method 1 ; F. Prieto 71.00%, (c) method 2 ; Lennetech 84.88%, (d) method 3 ; D.J Idiata 97.10%

A comparison of determination results has been made to verify the accuracy performance with commercial instrument ICP-OES. The results are presented in figure 10, a well-expected result is obtained by the Bland-Altman approach [48], two methods of measurement agree sufficiently closely. The measurement result from this instrument (experimental work) tightly scattered about the line of ICP measurement. Thus, the measurement are comparable.

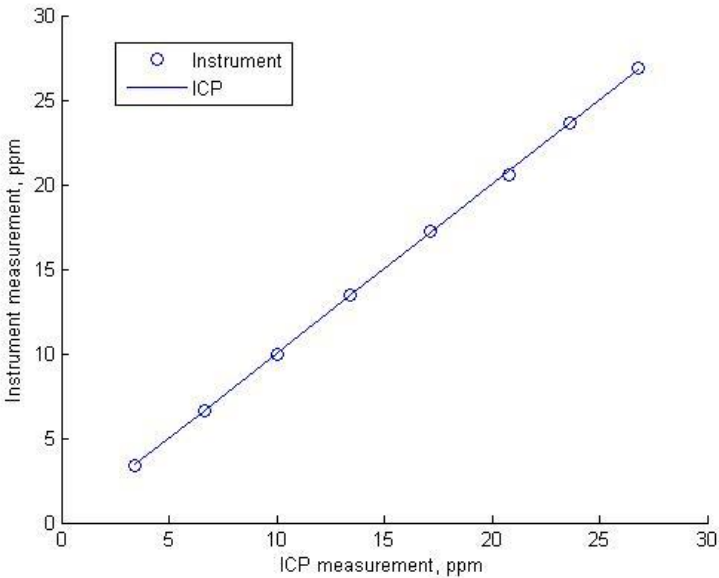


Figure 10: Comparing two measurement devices by Bland-Altman approach

Conclusion

These paper draws 4 main conclusions. Firstly, the presented method is greatly achieved to adapt in batik industry for monitoring wastewater status. A laboratory analysis for simulated batik wastewater using synthetic solutions was performed to analyze the performance of instrument development. The performance was increased by literature method comparison after consider a significant parameter. Secondly, mathematical formulation and algorithm was developed for determine wastewater status from the indication of conductivity, temperature and pH. The results are comparable to the commercial instrument, ICP-OES. Thirdly, data analysis and storage, transmission system through wireless network and graphical user interface was developed for installation at final discharge point and field application. At last, the integrating software and hardware has confirmed this instrument can be real-time monitoring, so the operator and employer can be instantaneously check and control the level of discharge effluent from the treatment and process.

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