



**Removal of Dyes from Industrial Effluents Using  
Combination of Advanced Oxidation Processes (AOPs)  
and Biological Treatment**

by

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## Removal of Dyes from Industrial Effluents Using Combination of Advanced Oxidation Processes (AOPs) and Biological Treatment

### ABSTRACT

Nowadays, the removal of dyes from industrial effluents is still far away to a satisfactory solution. Even though the AOPs are known strong technologies for wastewater treatment, it still requires further advancement and extent. Hence, a new promising treatment is their combination with biological treatment, by taking the advantages of the individual potentials. Therefore, this research evaluated four treatment techniques, namely ozonation, ozone/hydrogen peroxide ( $O_3/H_2O_2$ ), ultraviolet/hydrogen peroxide ( $UV/H_2O_2$ ), and a combination of ozonation-biological for synthetic dyes, consist of monoazo Methyl Orange (MO), disazo Reactive Red 120 (RR120) and anthraquinone Reactive Blue 19 (RB19). Finally, the treatments are evaluated with batik wastewater as a real wastewater sample from industries. The finding revealed that ozonation,  $O_3/H_2O_2$ ,  $UV/H_2O_2$ , and ozonation-biological become an effective treatment for monoazo, disazo, anthraquinone, and real wastewater. The treatments accomplish, under appropriate conditions, a full decolourization and a substantial mineralization. However,  $O_3/H_2O_2$  and ozonation works well with the dyes, in contrast to  $UV/H_2O_2$ . It reveals that complete decolourization by ozonation and  $O_3/H_2O_2$ , with less than 20 min contact. Two decolourization curves of ozonation and  $O_3/H_2O_2$  almost overlapped suggesting that  $H_2O_2$  hardly affects decolourization rate. Contrariwise, it takes more than 60 min for complete decolourization with  $UV/H_2O_2$  for RR120, but requires more than 120 min for MO and RB19. Nevertheless, there was a significant difference for COD and TOC removals. It is apparent that  $O_3/H_2O_2$  showed higher removal, suggesting that the presence of  $H_2O_2$  promote the oxidation reaction. The final COD removal of  $O_3/H_2O_2$  reached 100% within less than 10 min for RR120 and RB19, while 15 min for MO. Likewise, the higher TOC removal was observed for  $O_3/H_2O_2$  in comparison to ozonation and  $UV/H_2O_2$ . On the whole, the COD removal was similar to TOC removal for each treatment. It is obvious that high decolourization from the start of biological was contributed from ozonation pre-treatment. In addition, the results indicate that 59.6 and 69.4% COD removal from ozonation and ozonation-biological, respectively for MO. While, resulted about 40.7 and 72.9% removal for RR120, and 51.4 and 59.8% for RB19, respectively. Thus, it represents small organic molecules that contribute considerably to the COD that cannot be completely removed by ozonation-biological treatment. Similar to COD, the results indicate that 49.1 and 73.7% TOC removal from ozonation and ozonation-biological, respectively for MO. While it leads to 39.3 and 64.3% removal for RR120 and 37.5 and 70.8% removal for RB19, respectively. It is clear that the biological further degrades the dyes from ozonation. In addition, each dye shows different decolourization pattern and degradation behaviour according to its chemical structure. The change in UV-vis and FT-IR spectra indicated the evidence of dye structure cleavage and intermediates formation. While, the  $NO_3^-$ ,  $SO_4^{2-}$  and  $Cl^-$  anions formed indicate dye mineralization. The decolourization conform first-order kinetics, with  $R^2$  values greater than 0.92. The  $O_3/H_2O_2$  performs better with the batik wastewater, as compared to ozonation and  $UV/H_2O_2$ . Therefore, the results for synthetic wastewater support its application for real wastewater, even though the batik wastewater was more difficult to be decolourized and degraded because of its complex composition.

## Penyingkiran Pewarna daripada Efluen Perindustrian Menggunakan Gabungan Proses Pengoksidaan Lanjutan (PPL) dan Rawatan Biologi

### ABSTRAK

Pada masa kini, penyingkiran pewarna dari pelepasan efluen industri masih jauh lagi untuk mencapai penyelesaian yang memuaskan. Walaupun PPL dikenali sebagai teknologi yang baik untuk rawatan air sisa, ia masih lagi memerlukan penambahbaikan. Oleh itu, rawatan baru yang adalah gabungan PPL dengan rawatan biologi, dengan mengambil kira kelebihan potensi individu. Oleh itu, kajian ini dinilai empat teknik rawatan, iaitu pengozonan, ozon/hidrogen peroksida ( $O_3/H_2O_2$ ), ultraungu/hidrogen peroksida ( $UV/H_2O_2$ ), dan gabungan pengozonan-biologi pewarna sintetik, yang terdiri daripada *monoazo Methyl Orange* (MO), *disazo Reactive Red 120* (RR120) dan *anthraquinone Reactive Blue 19* (RB19). Akhir sekali, rawatan dinilai dengan air sisa batik sebagai sampel air sisa sebenar dari industri. Hasil kajian mendapati pengozonan,  $O_3/H_2O_2$ ,  $UV/H_2O_2$ , dan pengozonan-biologi menjadi satu rawatan berkesan untuk pewarna *monoazo*, *disazo*, *anthraquinone*, dan air sisa sebenar. Rawatan telah mencapai (dalam keadaan yang sesuai), penyingkiran penuh warna dan degradasi yang besar. Walau bagaimanapun,  $O_3/H_2O_2$  dan pengozonan berfungsi dengan lebih baik dengan pewarna, berbanding  $UV/H_2O_2$ . Ia menunjukkan bahawa penyingkiran sepenuhnya warna dengan pengozonan dan  $O_3/H_2O_2$  dalam masa kurang daripada 20 min. Dua lengkung penyingkiran warna daripada pengozonan dan  $O_3/H_2O_2$  hampir bertindih mencadangkan bahawa  $H_2O_2$  tidak memberi kesan kepada kadar penyingkiran warna. Sebaliknya, ia mengambil masa lebih daripada 60 minit untuk penyingkiran sepenuhnya dengan  $UV/H_2O_2$  untuk RR120, tetapi lebih daripada 120 min untuk MO dan RB19. Walau bagaimanapun, terdapat perbezaan yang signifikan untuk peyingkiran COD dan TOC. Ia adalah jelas bahawa  $O_3/H_2O_2$  menunjukkan penyingkiran yang lebih tinggi, dan kehadiran  $H_2O_2$  menggalakkan pengoksidaan. Penyingkiran COD akhir  $O_3/H_2O_2$  mencapai 100% dalam masa kurang daripada 10 minit untuk RR120 dan RB19, manakala 15 min untuk MO. Begitu juga, penyingkiran TOC yang lebih tinggi untuk  $O_3/H_2O_2$  berbanding pengozonan dan  $UV/H_2O_2$ . Pada keseluruhannya, penyingkiran COD adalah sama dengan TOC untuk setiap rawatan. Ia adalah jelas bahawa penyingkiran warna yang tinggi dari permulaan rawatan biologi disumbangkan dari pra-rawatan pengozonan. Di samping itu, keputusan menunjukkan bahawa 59.6 dan 69.4% penyingkiran COD dari pengozonan dan pengozonan-biologi, masing-masing untuk MO. Manakala, kira-kira 40.7 dan 72.9% untuk RR120, dan 51.4 dan 59.8% untuk RB19. Oleh itu, ia menunjukkan molekul organik kecil telah menyumbang dengan ketara kepada COD yang tidak boleh disingkirkan sepenuhnya oleh rawatan pengozonan-biologi. Sama seperti COD, keputusan menunjukkan bahawa 49.1 dan 73.7% penyingkiran TOC dari pengozonan dan pengozonan-biologi, masing-masing untuk MO. Walaupun, ia membawa kepada 39.3 dan 64.3% bagi RR120, dan 37.5 dan 70.8% bagi RB19. Ia adalah jelas bahawa rawatan biologi mendegradasikan lagi pewarna dari pengozonan. Selain itu, setiap pewarna menunjukkan corak yang berbeza mengikut struktur kimianya. Perubahan dalam spektrum UV-vis dan FT-IR menunjukkan bukti pemecahan struktur dan pembentukan produk perantaraan. Manakala, anion  $NO_3^-$ ,  $SO_4^{2-}$  dan  $Cl^-$  yang terbentuk menunjukkan degradasi pewarna. Penyingkiran warna menepati kinetik tertib-pertama, dengan nilai  $R^2$  yang lebih besar daripada 0.92.  $O_3/H_2O_2$  merawat air sisa batik dengan lebih baik, berbanding pengozonan dan  $UV/H_2O_2$ . Oleh itu, keputusan untuk air sisa sintetik menyokong penggunaan untuk air sisa sebenar, walaupun air sisa batik lebih sukar untuk penyingkiran warna dan degradasi disebabkan komposisinya yang lebih kompleks.

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1. Che Zulzikrami Azner Abidin, Fahmi, Ong Soon An, Siti Nurfatin Nadhirah Mohd Makhtar, Nazzery Rosmady Rahmat (2014). Decolorization and COD Reduction of Textile Wastewater by Ozonation in Combination with Biological Treatment. *Science of the Total Environment*. (*Impact Factor 3.789*) – *draft*
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## LIST OF SYMBOLS

|                        |  |
|------------------------|--|
| %                      | Percentage   |
| $^{\circ}\text{C}$     | Degree Celsius   |
| $a$                    | Weight concentration of $\text{H}_2\text{O}_2$   |
| $A$                    | Weight of dried filter plus dried residue  |
| Abs                    | Absorbance   |
| $B$                    | Weight of filter   |
| $C$                    | Concentration  |
| $C_0$                  | Concentration at time = 0  |
| $C_t$                  | Concentration at time = $t$  |
| $\text{cm}^{-1}$       | Wavenumber   |
| $d$                    | Dilution factor  |
| $D$                    | Chromogen  |
| $\text{g/mol}$         | Grams per mole   |
| $f$                    | Correction factor (ratio of the COD value of the $\text{H}_2\text{O}_2$ concentration) |
| $h$                    | Hour   |
| $\text{HO}^{\bullet}$  | Hydroxyl radicals  |
| $h\nu$                 | Photon   |
| $k$                    | Reaction rate constant   |
| $\lambda$              | Wavelength   |
| $\lambda_{\text{max}}$ | Maximum absorption wavelength  |
| $L$                    | Litre  |
| $lb$                   | Pound (mass)   |
| min                    | Minute   |
| $\text{mg/L}$          | Milligram per litre  |
| $\text{mL/min}$        | Millilitres per minute   |
| mM                     | Milimolar  |
| $N_{\text{MnO}_4}$     | Normality of $\text{KMnO}_4$ titrate   |
| nm                     | Nanometre  |
| $Q$                    | Linker or bridge   |
| $RG$                   | Reactive groups  |
| Pt-Co                  | Platinum-Cobalt Scale (colour scale)   |

|            |                                      |
|------------|--------------------------------------|
| $t$        | Time                                 |
| $T_{MnO4}$ | Volume of $KMnO_4$ titrate           |
| $\mu l$    | Microliter                           |
| $USD/m^3$  | United States dollar per cubic meter |
| $v$        | Volume                               |
| $V$        | Volts                                |
| $W$        | Water-solubilising group             |
| $W/m^2$    | Watts per square meter               |
| $X$        | Leaving group                        |

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## LIST OF ABBREVIATIONS

|   |                                      |
|---|--------------------------------------|
| ADMI  | American Dye Manufacturers Institute |
| Ag <sub>2</sub> SO <sub>4</sub>                 | Silver sulphate                      |
| AOPs  | Advanced oxidation processes         |
| AOX   | Absorbable organic halogens          |
| ATR   | Attenuated Total Reflection          |
| ASP   | Activated-sludge Process             |
| BOD   | Biochemical oxygen demand            |
| BOD <sub>5</sub>                                | Biochemical oxygen demand for 5 days |
| CAS   | Chemical Abstract Service            |
| CMAS  | Complete-mix activated sludge        |
| CI  | Colour Index                         |
| Cl  | Chlorine                             |
| Cl <sub>2</sub>                                 | Chlorine gas                         |
| ClO <sub>2</sub>                                | Chlorine dioxide                     |
| Cl <sup>-</sup>                                 | Chloride anions                      |
| ClO <sup>-</sup>                                | Hypochlorite                         |
| CMC   | Carboxymethyl cellulose              |
| COD   | Chemical oxygen demand               |
| COD <sub>c</sub>                                | Chemical oxygen demand (corrected)   |
| COD <sub>m</sub>                                | Chemical oxygen demand (measured)    |
| DO  | Dissolved oxygen                     |
| DOE   | Department of Environment            |
| EOP   | Electrochemical oxidation potential  |
| EQA   | Environmental Quality Act            |
| F   | Fluorine                             |
| FT-IR   | Fourier Transforms-Infrared          |
| GAC   | Granular activated carbon            |
| H <sub>2</sub> SO <sub>4</sub>                  | Sulphuric acid                       |
| HO <sup>•</sup>                                 | Hydroxyl radicals                    |
| H <sub>2</sub> O <sub>2</sub>                   | Hydrogen peroxide                    |
| H <sub>2</sub> O <sub>2</sub> /Fe <sup>2+</sup> | Fenton                               |

|   |   |
|---|---|
| H <sub>2</sub> SO <sub>4</sub>                | Sulphuric acid                                |
| HCl   | Hydrochloric acid                             |
| HgSO <sub>4</sub>                             | Mercury(II) sulphate                          |
| IC  | Ion-chromatography                            |
| ID  | Internal diameter                             |
| K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> | Potassium dichromate                          |
| KI  | Potassium iodide                              |
| MLSS  | Mixed liquor suspended solids                 |
| MLVSS   | Mixed liquor volatile suspended solids        |
| Mn <sub>2</sub> O <sub>7</sub>                | Manganese(VII)                                |
| Mo  | Microorganism                                 |
| MO  | Methyl Orange                                 |
| N <sub>2</sub>                                | Nitrogen gas                                  |
| Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> | Sodium thiosulphate                           |
| NaOH  | Sodium hydroxide                              |
| NBR   | Nitrile butyl rubber                          |
| NHAr  | Aromatic amines                               |
| NR  | Natural rubber                                |
| NRE   | Ministry of Natural Resources and Environment |
| NO <sub>3</sub> <sup>-</sup>                  | Nitrate anions                                |
| NO <sub>2</sub> <sup>-</sup>                  | Nitrite anions                                |
| O <sub>2</sub>                                | Oxygen (molecular)                            |
| O <sub>3</sub>                                | Ozone   |
| O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> | Ozone / Hydrogen peroxide, Perozone process   |
| PVA   | Polyvinyl alcohol                             |
| PVC   | Polyvinyl chloride                            |
| PU  | Polyurethane                                  |
| RB19  | Reactive Blue 19                              |
| rpm   | Revolution per minute                         |
| RR120   | Reactive Red 120                              |
| SBR   | Sequencing batch reactor                      |
| SO <sub>4</sub> <sup>2-</sup>                 | Sulphate anions                               |

|                                  |   |
|----------------------------------|---|
| TDS                              | Total Dissolved Solids  |
| TiO <sub>2</sub>                 | Titanium dioxide  |
| TOC                              | Total organic carbon  |
| TSS                              | Total suspended solids  |
| UBAF                             | Up-flow biological aerated filter   |
| UV                               | Ultraviolet   |
| UV/H <sub>2</sub> O <sub>2</sub> | Ultraviolet irradiation / Hydrogen Peroxide, H <sub>2</sub> O <sub>2</sub> photolysis process |
| UV-vis                           | Ultraviolet-visible   |
| VSS                              | Volatile suspended solids   |
| V-UV                             | Vacuum-ultraviolet  |
| ZnO                              | Zinc oxide  |

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## LIST OF FIGURES

| NO.  |  | PAGE |
|------|--|------|
| 1.1  | Compositions of water pollution sources by sector in Malaysia (2004)   | 3    |
| 1.2  | Typical steps involved in textile processing in cotton mill  | 5    |
| 2.1  | Molecular structure of Methyl Orange   | 19   |
| 2.2  | Molecular structure of Reactive Red 120  | 20   |
| 2.3  | Molecular structure of Reactive Blue 19  | 22   |
| 2.4  | Hydroxyl radical attack on aromatic compound   | 36   |
| 2.5  | O <sub>3</sub> molecules 1-3 dipolar Cyclo addition of direct reaction   | 41   |
| 3.1  | A2Z (model Z-3G) O <sub>3</sub> generator  | 64   |
| 3.2  | Schematic diagram of ozonation reactor: 1) O <sub>2</sub> cylinder, 2) flow meter, 3) O <sub>3</sub> generator, 4) glass reactor, 5) diffuser, 6) KI trap  | 65   |
| 3.3  | Ozonation and O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> reactor  | 66   |
| 3.4  | Schematic diagram of O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> reactor: 1) O <sub>2</sub> cylinder, 2) flow meter, 3) O <sub>3</sub> generator, 4) glass reactor, 5) diffuser, 6) KI trap, 7) H <sub>2</sub> O <sub>2</sub> addition | 67   |
| 3.5  | UV lamp model GPH 295T5L/4P  | 69   |
| 3.6  | Schematic diagram of UV/H <sub>2</sub> O <sub>2</sub> reactor: 1) light source, 2) UV lamp, 3) quartz sleeve, 4) water bath, 5) water jacket, 6) magnetic stirrer, 7) sampling port  | 70   |
| 3.7  | UV/H <sub>2</sub> O <sub>2</sub> reactor   | 71   |
| 3.8  | Schematic diagram of aerobic CMAS reactor: 1) air blower, 2) diffuser, 3) glass reactor, 4) paddle stirrer   | 73   |
| 3.9  | Complete-mix activated sludge (CMAS) reactor   | 73   |
| 3.10 | CMAS reactor operation cycle   | 74   |
| 3.11 | General flow chart of the experimental stages  | 89   |
| 4.1  | UV-vis spectra of (a) MO, (b) RR120 and (c) RB19 after ozonation at different contact time   | 93   |
| 4.2  | Colour removal after ozonation for MO, RR120 and RB19  | 95   |
| 4.3  | COD removal after ozonation for MO, RR120 and RB19   | 97   |
| 4.4  | TOC removal after ozonation for MO, RR120 and RB19   | 98   |
| 4.5  | FT-IR spectra of (a) 0 min, (b) 10 min and (c) 20 min ozonation for MO   | 100  |
| 4.6  | FT-IR spectra of (a) 0 min, (b) 10 min and (c) 20 min ozonation for RR120  | 101  |
| 4.7  | FT-IR spectra of (a) 0 min, (b) 10 min and (c) 20 min ozonation for RB19   | 102  |

|      |  |     |
|------|--|-----|
| 4.8  | Inorganic anions evolution during the ozonation of (a) MO, (b) RR120 and (c) RB19  | 105 |
| 4.9  | UV-vis spectra of (a) MO, (b) RR120 and (c) RB19 after O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> at different contact time | 107 |
| 4.10 | Colour removal after O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> for MO, RR120 and RB19                                      | 109 |
| 4.11 | Percentage removal of measured and corrected COD after O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> for MO                    | 110 |
| 4.12 | Percentage removal of measured and corrected COD after O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> for RR120                 | 111 |
| 4.13 | Percentage removal of measured and corrected COD after O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> for RB19                  | 112 |
| 4.14 | Corrected COD removal after O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> for MO, RR120 and RB19                               | 112 |
| 4.15 | TOC removal after O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> for MO, RR120 and RB19   | 113 |
| 4.16 | FT-IR spectra of (a) 0 min, (b) 10 min and (c) 20 min O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> for MO                     | 115 |
| 4.17 | FT-IR spectra of (a) 0 min, (b) 10 min and (c) 20 min O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> for RR120                  | 116 |
| 4.18 | FT-IR spectra of (a) 0 min, (b) 10 min and (c) 20 min O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> for RB19                   | 117 |
| 4.19 | Inorganic anions evolution during the O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> of (a) MO, (b) RR120 and (c) RB19          | 119 |
| 4.20 | UV-vis spectra of (a) MO, (b) RR120 and (c) RB19 after UV/H <sub>2</sub> O <sub>2</sub> at different contact time              | 122 |
| 4.21 | Colour removal after UV/H <sub>2</sub> O <sub>2</sub> for MO, RR120 and RB19   | 124 |
| 4.22 | Percentage removal of measured and corrected COD after UV/H <sub>2</sub> O <sub>2</sub> for MO                                 | 126 |
| 4.23 | Percentage removal of measured and corrected COD after UV/H <sub>2</sub> O <sub>2</sub> for RR120                              | 127 |
| 4.24 | Percentage removal of measured and corrected COD after UV/H <sub>2</sub> O <sub>2</sub> for RB19                               | 128 |
| 4.25 | Corrected COD removal after UV/H <sub>2</sub> O <sub>2</sub> for MO, RR120 and RB19  | 128 |
| 4.26 | TOC removal after UV/H <sub>2</sub> O <sub>2</sub> for MO, RR120 and RB19  | 129 |
| 4.27 | FT-IR spectra of (a) 0 min, (b) 10 min and (c) 20 min UV/H <sub>2</sub> O <sub>2</sub> for MO                                  | 131 |
| 4.28 | FT-IR spectra of (a) 0 min, (b) 10 min and (c) 20 min UV/H <sub>2</sub> O <sub>2</sub> for RR120                               | 132 |
| 4.29 | FT-IR spectra of (a) 0 min, (b) 10 min and (c) 20 min UV/H <sub>2</sub> O <sub>2</sub> for RB19                                | 133 |
| 4.30 | Inorganic anions evolution during the UV/H <sub>2</sub> O <sub>2</sub> of (a) MO, (b) RR120 and (c) RB19                       | 136 |
| 4.31 | MLSS concentration in CMAS for (a) MO, (b) RR120 and (c) RB19  | 138 |
| 4.32 | UV-vis spectra of CMAS for (a) MO, (b) RR120 and (c) RB19 without pre-treatment (0 min ozonation)                              | 140 |



|      |   |     |
|------|---|-----|
| 4.33 | UV-vis spectra of MO CMAS at different pre-treatment contact time of (a) 5 min, (b) 10 min, (c) 15 min and (d) 20 min   | 141 |
| 4.34 | UV-vis spectra of RR120 CMAS at different pre-treatment contact time of (a) 5 min, (b) 10 min, (c) 15 min and (d) 20    | 142 |
| 4.35 | UV-vis spectra of RB19 CMAS at different pre-treatment contact time of (a) 5 min, (b) 10 min, (c) 15 min and (d) 20 min | 143 |
| 4.36 | Colour removal after CMAS for (a) MO, (b) RR120 and (c) RB19 with different pre-treatment times                         | 145 |
| 4.37 | COD removal of CMAS for (a) MO, (b) RR120, and (c) RB19 with different ozonation pre-treatment times                    | 147 |
| 4.38 | COD removal of 30 days CMAS treatment with different pre-treatment times  | 148 |
| 4.39 | TOC removal after CMAS with 10 min ozonation pre-treatment for MO, RR120 and RB19                                       | 149 |
| 4.40 | FT-IR spectra of MO at 15 min ozonation pre-treatment in combination with CMAS  | 151 |
| 4.41 | FT-IR spectra of RR120 at 10 min ozonation pre-treatment in combination with CMAS                                       | 152 |
| 4.42 | FT-IR spectra of RB19 at 15 min ozonation pre-treatment in combination with CMAS  | 153 |
| 4.43 | Inorganic anions evolution during the ozonation in combination with CMAS of (a) MO, (b) RR120 and (c) RB19              | 155 |
| 4.44 | Proposed degradation pathway of MO  | 159 |
| 4.45 | Proposed degradation pathway of RR120   | 160 |
| 4.46 | Proposed degradation pathway of RB19  | 162 |
| 5.1  | Comparison of UV-vis spectra for (a) MO, (b) RR120 and (c) RB19 under ozonation, $O_3/H_2O_2$ and UV/ $H_2O_2$          | 164 |
| 5.2  | Comparison of decolourization profile for (a) MO, (b) RR120 and (c) RB19 under ozonation, $O_3/H_2O_2$ and UV/ $H_2O_2$ | 166 |
| 5.3  | Comparison of COD removal for (a) MO, (b) RR120 and (c) RB19 under ozonation, $O_3/H_2O_2$ and UV/ $H_2O_2$             | 167 |
| 5.4  | Comparison of TOC removal for (a) MO, (b) RR120 and (c) RB19 under ozonation, $O_3/H_2O_2$ and UV/ $H_2O_2$             | 168 |
| 5.5  | Comparison of UV-vis spectra of dyes with ozonation and without ozonation of (a) MO, (b) RR120 and (c) RB19             | 171 |
| 5.6  | Comparison of decolourization profiles of dyes with ozonation and without ozonation of (a) MO, (b) RR120 and (c) RB19   | 172 |

|      |  |     |
|------|--|-----|
| 5.7  | Comparison of COD removal of dyes with ozonation and without ozonation of (a) MO, (b) RR120 and (c) RB19   | 174 |
| 5.8  | Comparison of COD removal between ozonation and ozonation-CMAS   | 175 |
| 5.9  | Comparison of TOC removal between ozonation and ozonation-CMAS   | 176 |
| 6.1  | Evolution of pH before and after treatment during 10 min ozonation   | 179 |
| 6.2  | Effect of pH on the UV-vis spectra of: (a) MO, (b) RR120 and (c) RB19 after 10 min ozonation   | 180 |
| 6.3  | Effect of pH on the dye decolourization during 10 min ozonation  | 181 |
| 6.4  | Effect of pH on the COD concentration during 10 min ozonation  | 182 |
| 6.5  | Effect of initial dye concentration on the UV-vis spectra of: (a) 100, (b) 300 and (c) 500 mg/L MO during ozonation  | 184 |
| 6.6  | Effect of initial dye concentration on the UV-vis spectra of: (a) 100, (b) 300 and (c) 500 mg/L RR120 during ozonation   | 185 |
| 6.7  | Effect of initial dye concentration on the UV-vis spectra of: (a) 100, (b) 300 and (c) 500 mg/L RB19 during ozonation  | 186 |
| 6.8  | Effect of initial dye concentration on the colour removal of: (a) MO, (b) RR120 and (c) RB19 during ozonation  | 188 |
| 6.9  | Effect of initial dye concentration on the COD removal of: (a) MO, (b) RR120, and (c) RB19 during ozonation  | 189 |
| 6.10 | Evolution of pH before and after treatment during 10 min O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> with dosage of 0.67 mL H <sub>2</sub> O <sub>2</sub> /L dye                 | 191 |
| 6.11 | Effect of pH on the UV-vis spectra of: (a) MO, (b) RR120 and (c) RR19 with H <sub>2</sub> O <sub>2</sub> 0.67 mL/L dosage and 10 min O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> | 193 |
| 6.12 | Effect of pH on the dye decolourization during 10 min O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> with dosage of 0.67 mL H <sub>2</sub> O <sub>2</sub> /L dye                    | 194 |
| 6.13 | Effect of pH on the COD removal during 10 min O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> with H <sub>2</sub> O <sub>2</sub> 0.67 mL/L dosage                                    | 195 |
| 6.14 | Effect of H <sub>2</sub> O <sub>2</sub> dosage on the UV-vis spectra of: (a) MO, (b) RR120 and (c) RR19 after 10 min O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>                 | 197 |
| 6.15 | Effect of H <sub>2</sub> O <sub>2</sub> dosage on the decolourization of: (a) MO, (b) RR120, and (c) RB19 after 10 min O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>               | 199 |
| 6.16 | Effect of H <sub>2</sub> O <sub>2</sub> dosage on the COD removal after O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> of (a) MO, (b) RR120 and (c) RB19                            | 201 |
| 6.17 | Comparison of corrected COD removal with different H <sub>2</sub> O <sub>2</sub> dosage after 10 min O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>                                 | 202 |
| 6.18 | Effect of initial dye concentration on the UV-vis spectra of: (a) 100, (b) 300 and (c) 500 mg/L MO during O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>                            | 204 |

|      |   |     |
|------|---|-----|
| 6.19 | Effect of initial dye concentration on the UV-vis spectra of: (a) 100, (b) 300 and (c) 500 mg/L RR120 during O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>            | 205 |
| 6.20 | Effect of initial dye concentration on the UV-vis spectra of: (a) 100, (b) 300 and (c) 500 mg/L RB19 during O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>             | 206 |
| 6.21 | Effect of initial dye concentration on the colour removal of: (a) MO, (b) RR120 and (c) RB19 during O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>                     | 208 |
| 6.22 | Effect of initial dye concentration on the COD removal of: (a) MO, (b) RR120 and (c) RB19 during O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>                        | 210 |
| 6.23 | Evolution of pH before and after treatment during 60 min UV/H <sub>2</sub> O <sub>2</sub> treated with 0.67 ml/L H <sub>2</sub> O <sub>2</sub> dosage                 | 211 |
| 6.24 | Effect of pH on the UV-vis spectra of: (a) MO, (b) RR120 and (c) RR19 with 0.67 mL/L H <sub>2</sub> O <sub>2</sub> dosage and 60 min UV/H <sub>2</sub> O <sub>2</sub> | 213 |
| 6.25 | Effect of pH on the dye decolourization during 60 min UV/H <sub>2</sub> O <sub>2</sub> treated with 0.67 mL/L H <sub>2</sub> O <sub>2</sub> dosage                    | 214 |
| 6.26 | Effect of pH on the corrected COD removal during 60 min UV/H <sub>2</sub> O <sub>2</sub> treated with 0.67 mL/L H <sub>2</sub> O <sub>2</sub> dosage                  | 215 |
| 6.27 | Effect of H <sub>2</sub> O <sub>2</sub> dosage on the UV-vis spectra of: (a) MO, (b) RR120 and (c) RR19 after 60 min UV/H <sub>2</sub> O <sub>2</sub>                 | 218 |
| 6.28 | Effect of H <sub>2</sub> O <sub>2</sub> dosage on the decolourization of: (a) MO, (b) RR120, and (c) RB19 after 60 min UV/H <sub>2</sub> O <sub>2</sub>               | 220 |
| 6.29 | Effect of H <sub>2</sub> O <sub>2</sub> dosage on the COD removal of: (a) MO, (b) RR120 and (c) RB19 after UV/H <sub>2</sub> O <sub>2</sub>                           | 222 |
| 6.30 | Comparison of corrected COD removal with different H <sub>2</sub> O <sub>2</sub> dosage after 60 min UV/H <sub>2</sub> O <sub>2</sub>                                 | 223 |
| 6.31 | Effect of initial dye concentration on the UV-vis spectra of: (a) 100, (b) 300 and (c) 500 mg/L MO during UV/H <sub>2</sub> O <sub>2</sub>                            | 225 |
| 6.32 | Effect of initial dye concentration on the UV-vis spectra of: (a) 100, (b) 300 and (c) 500 mg/L RR120 during UV/H <sub>2</sub> O <sub>2</sub>                         | 227 |
| 6.33 | Effect of initial dye concentration on the UV-vis spectra of: (a) 100, (b) 300 and (c) 500 mg/L RB19 during UV/H <sub>2</sub> O <sub>2</sub>                          | 228 |
| 6.34 | Effect of initial dye concentration on the colour removal of: (a) MO, (b) RR120, and (c) RB19 during UV/H <sub>2</sub> O <sub>2</sub>                                 | 230 |
| 6.35 | Effect of initial dye concentration on the COD removal of: (a) MO, (b) RR120 and (c) RB19 during UV/H <sub>2</sub> O <sub>2</sub>                                     | 231 |
| 7.1  | Comparison of dye decolourization kinetics with ozonation   | 234 |
| 7.2  | Comparison of dye decolourization kinetics with O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>   | 234 |
| 7.3  | Comparison of dye decolourization kinetics with UV/H <sub>2</sub> O <sub>2</sub>  | 234 |

|      |  |     |
|------|--|-----|
| 8.1  | Comparison of UV-vis spectra for batik wastewater after ozonation, O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> and UV/H <sub>2</sub> O <sub>2</sub>        | 239 |
| 8.2  | Comparison of discoloration profile for batik wastewater under ozonation, O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> and UV/H <sub>2</sub> O <sub>2</sub> | 240 |
| 8.3  | Comparison of COD removal for batik wastewater under ozonation, O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> and UV/H <sub>2</sub> O <sub>2</sub>           | 241 |
| 8.4  | Comparison of TOC removal for batik wastewater under ozonation, O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub> and UV/H <sub>2</sub> O <sub>2</sub>           | 242 |
| 8.5  | MLSS concentration during ozonation-CMAS of batik wastewater   | 244 |
| 8.6  | UV-vis spectra of CMAS treatment for batik wastewater without ozonation  | 245 |
| 8.7  | UV-vis spectra of batik wastewater CMAS treatment at different ozonation contact time of (a) 15 min, (b) 30 min, (c) 45 min and (d) 60 min                   | 246 |
| 8.8  | Comparison of UV-vis spectra of batik wastewater with ozonation and without ozonation  | 247 |
| 8.9  | Comparison of decolourization patterns of batik wastewater with ozonation and without ozonation  | 248 |
| 8.10 | Comparison of COD removal of batik wastewater with ozonation and without ozonation   | 249 |
| 8.11 | TOC concentration of (a) ozonation and (b) ozonation-CMAS treatment of batik wastewater  | 250 |
| 8.12 | Comparison of COD and TOC removal between ozonation-CMAS treatment batik wastewater  | 251 |

## LIST OF TABLES

| NO. |  | PAGE |
|-----|--|------|
| 2.1 | Global market shares of different dye classification   | 15   |
| 2.2 | Example classification of common dyes based on the chemical structure                                  | 17   |
| 2.3 | Typical characteristics of textile wastewater  | 25   |
| 2.4 | Conventional methods for removal of dye-containing wastewater  | 26   |
| 2.5 | Comparison of oxidation potential of various oxidants  | 37   |
| 2.6 | Non-photochemical and photochemical methods of AOPs  | 38   |
| 3.1 | Properties and characteristics of the studied reactive dyes  | 59   |
| 3.2 | Physical parameters of the studied batik wastewater  | 61   |
| 3.3 | List of chemicals and reagents   | 62   |
| 7.1 | Comparison of reaction rate and half-life decolourization of the synthesized dye-containing wastewater | 235  |

## TABLE OF CONTENTS

|   | <b>PAGE</b> |
|---|-------------|
| <b>DECLARATION</b>                          | ii          |
| <b>DEDICATION</b>                           | iii         |
| <b>ACKNOWLEDGEMENT</b>                      | iv          |
| <b>TABLE OF CONTENTS</b>                    | vi          |
| <b>LIST OF TABLES</b>                       | xii         |
| <b>LIST OF FIGURES</b>                      | xiii        |
| <b>LIST OF ABBREVIATION</b>                 | xix         |
| <b>LIST OF SYMBOLS</b>                      | xxii        |
| <b>ABSTRAK</b>                              | xxiv        |
| <b>ABSTRACT</b>                             | xxv         |
| <br>  |             |
| <b>CHAPTER 1 INTRODUCTION</b>               |             |
| 1.1 Environmental Pollutions                | 1           |
| 1.2 Industrial Wastewater Pollutions        | 2           |
| 1.3 Dyes Treatment Methods                  | 7           |
| 1.4 Problem Statements                      | 8           |
| 1.5 Research Objectives                     | 11          |
| 1.6 Research Scope                          | 12          |
| 1.7 Thesis Outline                          | 13          |
| <br>  |             |
| <b>CHAPTER 2 LITERATURE REVIEW</b>          |             |
| 2.1 Introduction                            | 14          |
| 2.2 Dye Classification                      | 16          |
| 2.2.1 Azo Dyes                              | 18          |
| 2.2.1.1 Acid Orange 52 / Methyl Orange (MO) | 18          |
| 2.2.1.2 Reactive Red 120 (RR120)            | 19          |
| 2.2.2 Anthraquinone Dyes                    | 20          |
| 2.2.2.1 Reactive Blue 19 (RB19)             | 21          |
| 2.3 Dye Structure                           | 22          |
| 2.4 Dye, and the Environmental Concern      | 23          |

|         |   |    |
|---------|---|----|
| 2.5     | Dye Removal Method                                | 25 |
| 2.5.1   | Biological Treatment                              | 28 |
| 2.5.1.1 | Anaerobic Process                                 | 30 |
| 2.5.1.2 | Aerobic Process                                   | 30 |
| 2.5.2   | Physical Treatment                                | 32 |
| 2.5.3   | Chemical Treatment                                | 33 |
| 2.6     | Advanced Oxidation Processes (AOPs)               | 35 |
| 2.6.1   | Ozonation   | 39 |
| 2.6.1.1 | Fundamental                                       | 40 |
| 2.6.1.2 | Advantages and Disadvantages                      | 42 |
| 2.6.1.3 | Application to Dye-containing Wastewater          | 43 |
| 2.6.2   | O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>     | 45 |
| 2.6.2.1 | Fundamental                                       | 45 |
| 2.6.2.2 | Advantages and Disadvantages                      | 46 |
| 2.6.2.3 | Application to Dye-containing Wastewater          | 47 |
| 2.6.3   | UV/H <sub>2</sub> O <sub>2</sub>                  | 48 |
| 2.6.3.1 | Fundamental                                       | 48 |
| 2.6.3.2 | Advantages and Disadvantages                      | 49 |
| 2.6.3.3 | Application to Dye-containing Wastewater          | 50 |
| 2.7     | Combination of Ozonation and Biological Treatment | 52 |
| 2.7.1   | Fundamental                                       | 53 |
| 2.7.2   | Advantages and Disadvantages                      | 54 |
| 2.7.3   | Application to Dye-containing Wastewater          | 54 |
| 2.8     | Treatment of Batik Wastewater                     | 56 |

### **CHAPTER 3 MATERIALS AND METHODOLOGY**

|       |   |    |
|-------|---|----|
| 3.1   | Introduction                                    | 58 |
| 3.2   | Materials                                       | 58 |
| 3.2.1 | Dyes  | 58 |
| 3.2.2 | Batik Wastewater                                | 60 |
| 3.2.3 | Chemicals and Reagents                          | 61 |
| 3.3   | Synthetic Dye-containing Wastewater Preparation | 62 |

|         |   |    |
|---------|---|----|
| 3.4     | Reactor and Experimental Procedures                 | 63 |
| 3.4.1   | Ozonation   | 64 |
| 3.4.1.1 | Reactor Set-up                                      | 64 |
| 3.4.1.2 | Experimental Procedure                              | 65 |
| 3.4.2   | O <sub>3</sub> /H <sub>2</sub> O <sub>2</sub>       | 67 |
| 3.4.2.1 | Reactor Set-up                                      | 67 |
| 3.4.2.2 | Experimental Procedure                              | 68 |
| 3.4.3   | UV/H <sub>2</sub> O <sub>2</sub>                    | 69 |
| 3.4.3.1 | Reactor Set-up                                      | 69 |
| 3.4.3.2 | Experimental Procedure                              | 71 |
| 3.4.4   | Combination of Ozonation and Biological Treatment   | 72 |
| 3.4.4.1 | Complete-mix Activated Sludge (CMAS) Reactor Set-up | 72 |
| 3.4.4.2 | Experimental Procedure                              | 74 |
| 3.5     | Analytical Procedure                                | 76 |
| 3.5.1   | UV-vis Spectrophotometer                            | 76 |
| 3.5.2   | Ozone (O <sub>3</sub> ) Flow Rate                   | 77 |
| 3.5.3   | H <sub>2</sub> O <sub>2</sub> Residual              | 78 |
| 3.5.4   | Chemical Oxygen Demand (COD)                        | 80 |
| 3.5.4.1 | Measured COD  | 80 |
| 3.5.4.2 | Corrected COD                                       | 81 |
| 3.5.5   | Total Organic Carbon (TOC)                          | 83 |
| 3.5.6   | Fourier Transform-Infrared (FT-IR) Spectroscopy     | 84 |
| 3.5.7   | Ion Chromatography (IC)                             | 85 |
| 3.5.8   | Mixed Liquor Suspended Solids (MLSS)                | 86 |
| 3.6     | Decolourization Kinetics                            | 87 |

## **CHAPTER 4 PERFORMANCE EVALUATION ON THE COLOUR, COD, TOC AND CONTAMINANTS CHARACTERIZATION**

|       |                           |    |
|-------|---------------------------|----|
| 4.1   | Introduction              | 90 |
| 4.2   | Ozonation                 | 92 |
| 4.2.1 | UV-vis Absorption Spectra | 92 |
| 4.2.2 | Colour Removal            | 94 |