A Review of 2,4-Dichlorophenoxyacetic Acid (2,4-D) Derivatives: 2,4-D Dimethylamine Salt and 2,4-D Butyl Ester

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Abstract
2,4-dichlorophenoxyacetic acid (2,4-D) and its derivatives are one of the most used herbicides in the world. They have been used since 1940s and their popularity seems to be increasing. Due to their broad range of application and excessive usage, they have becoming a serious problem as they can cause pollution to the environment the environment. Extensive researches have been done for 2,4-D but comparatively few for its derivatives. Therefore, In this review paper, we focused more on 2,4-D derivatives including 2,4-D dimethylamine salt and 2,4-D butyl ester in order to have better understanding on the fates of this pesticide in the environment. Hence, their physical and chemical properties are reviewed based on available references. In this paper, we also discussed the toxicity effects of 2,4-D and its derivatives on the environment. This review also described about the treatment technologies for 2,4-D and its derivatives.

Keywords: herbicides, 2,4-D and its derivatives, environmental fate, toxicity, treatment technology

INTRODUCTION
Application of herbicides is one of the general agricultural practice for maintaining high productivity and plant’s growth. They are not only used in agriculture sector but also for other applications such as home lawn maintenance, gardening, golf course, parks and forestry. Herbicides is commonly used to kill or remove any type of pests. Other chemicals that are categorize as pesticides are fungicides, insecticides, rodenticides, nematicides, termicidites and molluscicides. Herbicide is defined as a group of chemicals known as plant protection products (PPP) that used to kill or repel any type of weedy plant in order to protect the crop [1]. Herbicides help to manage weed problem effectively and economically. Herbicides can be classified according to its mode and site action. For example, the mode of action for 2,4-dichlorophenoxyacetic acid (2,4-D) and its formulation are plant growth regulators which mean that they will upsets normal plant growth [2]. Herbicides are commonly formulated in two forms including sprayed liquid and dry solids. Herbicides are widely used all over the world. Fig.1. shows the statistic of pesticide usage worldwide, according to its category for the year of 2011 [3]. The total usage of herbicides in worldwide accounted as 36% followed by insecticides (25%), fungicides (10%) and other pesticides including nematicides, rodenticides, fumigants, bird, fish and aquatic fish (29%).

Figure 1. Pesticides Usage Worldwide

Among these herbicides, 2,4-dichlorophenoxyacetic acid or known as 2,4-D are one of the most widely used herbicide in the world. 2,4-D also come in the form of free acid, alkaline and amine salts and ester formulations [4]. 2,4-D and its derivatives are created according to their specific function. They are one of the phenoxy or phenoxyacetic acid herbicide family which belong to one of the largest herbicides group [5]. These type of herbicides family are popular among the farmers because of their low cost, effectiveness even in low doses and good water solubility [6]. They are designed to play the same role as natural auxin, indole-3-yl-acetic acid which vital in the division, differentiation and elongation of plant cells [7]. Therefore, they can disrupt the plant growth naturally. Based on TABLE.1., 2,4-D are also known as the plant growth regulator. There are other herbicide families that have the same role of 2,4-D such as MCPA, benzoic acid, phthalamates and many more [2].
The chemical name for 2,4-D is 2,4-dichlorophenoxyacetic acid is a white crystalline solid with chemical formula of C₈H₈Cl₂O₂. The molecular weight for 2,4-D is 221.0 g/mole [22]. 2,4-D is a non-volatile acid and was classified as Category D carcinogen [23]. 2,4-D n-butyl ester are one of the 2,4-D formulation in form of ester including 2,4-D 2-butoxylethyl ester (BEE), 2,4-D 2-ethylhexyl (2-EHE) and 2,4-D isopropyl ester (IPE). 2,4-D esters will hydrolyzed rapidly into free acid. 2,4-D n-butyl ester was used as an Agent Orange which is a mixture of 2,4-D and 2,4,5-T that used as a spray defoliant during Vietnam War [4]. 2,4-D n-butyl ester or known as 2,4-D BE is a high volatile ester form of 2,4-D with a chemical formula of C₁₂H₁₄Cl₂O₂. The molecular weight is 277.15 g/mol. The vapor pressure of 5.29X10⁻² to 8.2X10⁻¹ Pa indicate that this ester can volatilize from soil and crop surfaces. The Henry’s Law for 2,4-D BE is 4.88X10⁻⁷ atm.m³/mole show that 2,4-D BE may be moderately volatile to volatile from water bodies. The ester is soluble in organic solvent and insoluble in water [23].

One of the 2,4-D formulation in salt is 2,4-D dimethylamine salt followed by 2,4-D sodium salt (Na), 2,4-D diethanolamine salt (DEA), 2,4-D isopropylamine salt (IPA) and 2,4-D triisopropanolamine salt (TIPA). 2,4-D dimethylamine salt is an aquatic herbicide and it is use for control of broadleaf weeds.
in non-crop areas, lawns, ponds, ditch banks, pastures, rangelands and also for control of trees by injection [22]. 2,4-D dimethylamine salt or 2,4-D DMA with a chemical formula of C<sub>10</sub>H<sub>12</sub>ClN<sub>3</sub>O<sub>2</sub> is one of 2,4-D salt formulation. The molecular weight of 2,4-D DMA is 266.13 g/mol. It comes in form of aqueous liquid with amber color [22]. According to report [22], it will dissociates to acid when in water. The herbicides is non-volatile and soluble in water [23]. TABLE.2. explains more detail about the chemical and physical properties of 2,4-D and its form. These properties determine their behavior in the environment.

Table 2. Chemical and physical properties of 2,4-D and its derivatives

<table>
<thead>
<tr>
<th>Properties</th>
<th>2,4-D acid</th>
<th>2,4-D dimethylamine salt</th>
<th>2,4-D butyl ester</th>
</tr>
</thead>
<tbody>
<tr>
<td>CAS No.</td>
<td>94-75-7</td>
<td>2008-39-1</td>
<td>94-80-4</td>
</tr>
<tr>
<td>Melting point (25°C)</td>
<td>138 – 141</td>
<td>118 – 120</td>
<td>9</td>
</tr>
<tr>
<td>Relative Density</td>
<td>1.416</td>
<td>1.1502</td>
<td>1.246</td>
</tr>
<tr>
<td>Log K&lt;sub&gt;ow&lt;/sub&gt; (25°C)</td>
<td>pH 1 = 2.70</td>
<td>pH 5 = 0.18</td>
<td>pH 7 = 0.83</td>
</tr>
<tr>
<td>Solubility in water (mg/L)</td>
<td>569 (20°C)</td>
<td>72.9x10&lt;sup&gt;4&lt;/sup&gt; (20°C)</td>
<td>46</td>
</tr>
<tr>
<td>Vapor pressure (mm Hg, 25°C)</td>
<td>1.4x10&lt;sup&gt;-7&lt;/sup&gt;</td>
<td>&lt;1x10&lt;sup&gt;-7&lt;/sup&gt;</td>
<td>3.97x10&lt;sup&gt;-4&lt;/sup&gt;</td>
</tr>
<tr>
<td>Henry’s law constant (atm.m&lt;sup&gt;3&lt;/sup&gt;/mol)</td>
<td>3.54x10&lt;sup&gt;-8&lt;/sup&gt;</td>
<td>Dissociates into acid in water</td>
<td>4.88x10&lt;sup&gt;-7&lt;/sup&gt;</td>
</tr>
<tr>
<td>pKa</td>
<td>2.73</td>
<td>2.6</td>
<td>na</td>
</tr>
</tbody>
</table>

Octanol-water partition coefficient (K<sub>ow</sub>); dissociation constant (pKa); not available (na)

ENVIRONMENTAL FATE
Herbicides can contaminate our environment bodies including water, soil and air after their application. Due to their wide usage, human and other organisms also exposed to herbicides. When they come in contact with the environment, they will undergo various processes that will determine their fate and persistency in the environment depending on chemical, biological and physical properties of the herbicides itself and also the environment. Fig.3. showed the environmental fate of herbicides in the environment. The herbicides will undergo various processes such as photodegradation, microbial degradation and chemical degradation that determine their persistency in the environment or travel to every part of the environment via runoff, leaching, volatilization and many other processes [24].

Environmental fate of 2,4-D dimethylamine salt (2,4-D DMA) and 2,4-D n-butyl ester (2,4-D BE) has been studied for long term but there is limited information about their behavior in the environment. Hydrolysis is one of the chemical degradation processes that involve a reaction with hydrogen and it is usually in the form of water [25]. Hydrolysis process of 2,4-D esters has been studied under laboratory conditions and it is shown that the rate of this process is depending on the alcohol function of ester [26-28]. 2,4-D BE is proved to be hydrolyzed completely within 24 hours at condition of soil moisture above the wilting point [28]. A study found out that 2,4-D BE also hydrolyzed rapidly in the Mahloon soil and the half-life for the process is 26 minutes [23]. It is revealed that 2,4-D BE hydrolyzed quickly under acid condition according to rapid change in the rate of hydrolysis process. A study of [28] reported that 2,4-D DMA also dissociate to 2,4-D acid after its application in the soil and this finding. Wilson et al. found out that the rate of 2,4-D dissipation in soil is equivalent for both 2,4-D dimethylamine salt and 2,4-D 2-ethylhexyl ester.

Leaching is another process that determines the fate of these herbicides is the soil. Leaching is happened when the water is move downward in the soil through rain or irrigation water and caused the pollutants to move across the soil. One of the factors that affected the leaching process is soil texture and structure [29] The leaching process can cause water pollution as this process can contaminate the groundwater [30-31]. A study of 2,4-D DMA leaching process is carried out by [32] revealed that the amount of recovered 2,4-D DMA in the leachate is higher from soil column under the infrequent irrigation compare to soil column from frequent irrigation regime. This indicates that the leaching process is influence by the irrigation frequency. 2,4-D DMA is also found out to be degraded during this 28 days of test period due to only 3% of recovered 2,4-D DMA amount found in the leachate, soil layers and thatch layer [32].

One of the important processes that determine the persistency of herbicides in the environment is the photodegradation process. It is one of the degradation process including microbial and chemical degradation. Photodegradation process is divided into four main type which are direct photodegradation, photosensitized degradation, photocatalyzed degradation and degradation by reaction with hydroxyl radical [33]. The evaluation of 2,4-D DMA based on photocatalyzed degradation by using zinc oxide as the catalyst showed that the photodegradation process is influence by initial pH which is
acids, ZnO concentration and recycling flow rate in a flat-plate reactor [34]. The use of radiation chemistry in degradation process of organic pollutants in water is also important to protect the environment. The degradation of 2,4-D DMA by gamma radiation from cobalt-60 in water was successful at higher doses of 3 kGy and the produce metabolite is 4,6-dichlororesorcinol. It was revealed that the process is relied on the radiation dose and also on its degradation product [35].

TOXICITY OF 2,4-D AND ITS DERIVATIVES

A. Toxicity level of 2,4-D in the environment

Many researches have been conducted to detect the presence of 2,4-D in the environment. 2,4-D have been found in all over the world due to its widespread use as a herbicide. TABLE 3. presents some of the founding about the concentration level of 2,4-D in the environment. Mostly 2,4-D is detected in the water source such as the river, pond, ground water and others. This contamination is caused by the increasing amount of 2,4-D application and also improper way of disposal wastewater into the environment [36]. Besides, 2,4-D also can pollute the water source directly due to its application as aquatic weeds herbicide [37]. 2,4-D can reached the surface and ground water by runoff process due to its low soil adsorptivity, high potential of leachability and high water solubility [38-39]. The low biodegradability of 2,4-D in the water also make 2,4-D as the major pollutant in the water sources surrounding [40]. 2,4-D also can be detected in the human semen and urine sample. Living life such as humans and animals are exposed to 2,4-D by the contaminated air, drinking water, soil and foods if the human works in the agriculture sector and in the factory that produce 2,4-D [4].

B. Toxicity effect of 2,4-D and its derivatives to living life

Toxicity of pesticides can be classified into neurotoxicity, genotoxicity, cytotoxicity, hepatotoxicity and many more. Toxicity can be defined as negative effect of certain substance that capable of damaging the structure or any processes which are vital for organism survival [46]. Factors that influence the pesticide toxicity are concentration, frequency, intensity of exposure and target organism susceptibility, which is depend on age, sex, health state and genetic variations [47-48]. Among pesticides, phenoxyacetic acid herbicides showed hepatotoxic and nephrotoxic effects in animal studies when they are exposed to high level of these herbicides.

Table 3. Concentration level of 2,4-D in the environment

<table>
<thead>
<tr>
<th>Compound</th>
<th>Source</th>
<th>Concentration level</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,4-D</td>
<td>Semen</td>
<td>≥ 5.0 ng/mL</td>
<td>[41]</td>
</tr>
<tr>
<td>Stormwater</td>
<td>retention ponds in Regina, Canada</td>
<td>1204 ~ 2755 ng/L</td>
<td>[42]</td>
</tr>
<tr>
<td>Tap water</td>
<td></td>
<td>16.4 µg/L</td>
<td>[43]</td>
</tr>
<tr>
<td>River water, North of Iran</td>
<td></td>
<td>16.6 µg/L</td>
<td>[43]</td>
</tr>
<tr>
<td>Fish farming water, North of Iran</td>
<td></td>
<td>14.9 µg/L</td>
<td>[43]</td>
</tr>
<tr>
<td>River Ebro Basin in La Rioja</td>
<td></td>
<td>0.02 – 0.17 µg/L</td>
<td>[44]</td>
</tr>
<tr>
<td>Ground water in Ireland</td>
<td></td>
<td>0.001 µg/L</td>
<td>[1]</td>
</tr>
<tr>
<td>Urine sample</td>
<td></td>
<td>1.80 µg/L</td>
<td>[45]</td>
</tr>
</tbody>
</table>

Researcher found out that they might cause human chronic liver injury, non-Hodgkin’s lymphoma, soft tissue sarcoma and malignant lymphoma. 2,4-D and its derivatives are one of the phenoxyacetic acid herbicides that is highly concern for their strong genotoxic effects in different type of organism [49]. A few studies have been done to study the toxicity behavior of 2,4-D dimethylamine salt and 2,4-D n-butyl ester in the animals. Study of [50] has proved that 2,4-D DMA can caused development toxicity on rat’s infants. The salt can give negative effect on dental development of rat’s infant when they feed 2,4-D DMA to pregnant rat even in low doses and also can caused vitamin A deficiency for formation of dentin and enamel. This study has concluded that besides chlorinated organic compounds, dioxins and furans, chlorinated organic pesticides may also affect the dental development of infant when taken via mother’s milk [50]. 2,4-D DMA also shown a hypoglycemic effect in rats and a hyperglycemic effect in cattle [51-53]. Report [53] had revealed that 2,4-D DMA can decrease glucose level in mice’s blood but there is no change in activity of enzyme a amylase or liver function test. This study however proved that 2,4-D DMA could give a hypoglycemic effect on mice [53].

In addition, 2,4-D DMA can caused acute poisoning on rat and several damage on lymphatic organs such as spleen and thymus [54]. 2,4-D DMA also showed its ecotoxic effect on rats by decreasing rat’s body weight, liver and pancreas when they are fed by raw wastewater from manufacturing of 2,4-D DMA [55]. Experiment [55] concluded that 2,4-D DMA may initiate cancer on rats due to possibility of transformation of adenoma or carcinoma by neoplastic variations caused by this wastewater during long-term treatment according to observation of dilation in sinusoids close to the vein centralis and hydropic degeneration in parenchyma. Due to high volatility of 2,4-D n-butyl ester, it is often formulated as sprayed liquid and it is always use in aerial spraying. This situation give negative impact on different type of animals and avian eggs by exposing them to 2,4-D BE directly or indirectly. Report [56] shown that 2,4-D BE is embryotoxic when newly hatched chicks from these contaminated eggs experienced postural and motor dysfunctions, curled claws and other negative effects. 2,4-D BE is detected on 5th day of embryo development and the amount is increased as the embryo develop into hatched chick. All the analyzed organs and tissues of hatched chicks showed the presence of this ester and the highest level is found in brain and kidney [56].

Since these herbicides are mostly used in the agriculture sector, the agricultural workers will expose to these herbicides either directly or indirectly. Even though they are wearing personal protective equipment, there are still some risk due to prolonged exposure toward these herbicides. 2,4-D DMA exposure to the agricultural workers during occupational periods had caused the thioethers concentrations in their urine increases [57]. The glucose level in blood also decreases during the exposure of...
2,4-D DMA for 2 to 4 days working periods [58,53]. 24-D DMA showed genotoxicity and cytotoxicity effect in human red blood cells by inflicted the DNA and cellular damage. 2,4-D DMA also a potent genotoxic agent in the blood cells [59].

**TREATMENT TECHNOLOGIES FOR 2,4-D**

There are many methods that have been developed by the scientist to remove the presence of 2,4-D in the water regardless of their sources. This discussion focus on treatment of 2,4-D only due to limited source of reference regarding its other salt and ester forms. However, for 2,4-D DMA the treatment has been discussed in another section which is in the environment fate of herbicides. Basically, the treatments can be divided into two main groups which are destructive processes such as oxidation and non-destructive processes like adsorption [60]. Table 4 describes a few examples of the single and combined treatment that have been studied include advanced oxidation processes (AOPs) [40,61], adsorption process [62], biological process [63,64] electrochemical technology [65] and ionizing radiation [66]. Each of the individual treatment has its own advantages and disadvantages. Factors such as the treatment effectiveness, expenditure costs, exposure time, chemical stability and others play a vital role in choosing the suitable treatment.

One of the common treatment used to treat 2,4-D in the water is advanced oxidation processes (AOPs). AOPs is based on the oxidation process which produce free radicals (OH) that can destroy the organic pollutant through a sequence of reactions [40]. Examples of AOPs that can produce free radicals are photocatalysis process, heterogeneous catalysis, ozone and hydrogen peroxide-related processes, Fenton-like processes and many more [60]. Photocatalysis is one of the AOPs method that used a photocatalyst to degrade the 2,4-D under different source of light such as ultraviolet (UV) or visible light. This method highly dependent of different type of photocatalyst, source of light, initial concentration of 2,4-D and the pH solution [36,40,67-68]. Titanium dioxide (TiO$_2$) is the most common semiconductor used as a photocatalyst which possess a good resistance to photo-corrosion, photostability, low cost, non-toxicity and has high photocatalytic activity [40]. To enhance the photocatalytic activity, TiO$_2$ can be supported by material like zeolites or doping with nitrogen and other material like non-metal doped material (e.g. N,S co-doped nano titania) [36,40,68]. There is also a new mesoporous catalyst name as Si-CuF$_{16}$Pe catalyst that have been invented to follow the trend of green chemical method in order to undergo an environmentally friendly process [67]. AOPs method can also be combine with different type of processes such as ozone with ultraviolet radiation, ozone with hydrogen peroxide, ozone/hydrogen peroxide with ultraviolet radiation and so on. Combination method like ozone-hydrogen peroxide process have been studied by [69] is depend on the H$_2$O$_2$/O$_3$ molar ratio and pH value. Another example of combine process is UV irradiation with Fenton reagent (Fe$_{2+}$/H$_2$O$_2$/UV) or cobalt/peroxymonosulfate (Co/PMS/UV) have been done by Erick et al. 2007. This experiment shows that the degradation process depend on the concentration of cobalt and Fe(II) and the solar light as the source of light energy [70]. Besides AOPs, oxidation process with ammonium persulfate is considered as an alternative method to AOPs due to the production of persulfate radical (S$_2$O$_8^{2-}$) which offer more advantages such as good chemical stability and good oxidizing strength compare to hydroxyl radical [60].

<table>
<thead>
<tr>
<th>Table 4. Treatment technology for removal of 2,4-D</th>
</tr>
</thead>
<tbody>
<tr>
<td>Treatment</td>
</tr>
<tr>
<td>---</td>
</tr>
<tr>
<td>Photocatalytic degradation</td>
</tr>
<tr>
<td>Photo-Fenton process</td>
</tr>
<tr>
<td>Adsorptive removal</td>
</tr>
</tbody>
</table>
### Biological process

- **Use a microbial community of 2,4-D degrading in continuously operated packed-bed column (PBC)**
- **Initial concentration = 27.7 ppm**
- **Aeration rate = 0.1 L/min**
- **Temperature = 25 °C**
- **Duration = 150 days**
- **91.75%**

### Ionizing radiation

- **Use γ-radiation from 60Co- γ-source to decompose 2,4-D in presence of air**
- **Source dose = 4kGy**
- **Initial concentration = 500 µM**
- **pH = 9**
- **Complete removal**
- **2,4-DCP, isomers of phenoxyacetic acid and hydroxylation products of 2,4-D**

### Sono-electrochemical Fenton (SEF) process

- **Using a combination of ultrasound waves, electrochemistry and Fenton’s reagent**
- **Initial concentration = 1.2 mM**
- **pH = 3**
- **duration = 60 s**
- **50% oxidation of 2,4-D**
- **Formate, lactone, 2,4-DCP, quinone, diphenoxyethane**

### Combined process

- **Use an electrochemical flow cell for pre-treatment and biological treatment using activated sludge**
- **Initial concentration = 500 ppm**
- **pH = 7**
- **duration = 7 days**
- **96%**
- **Chlorohydroquinone**

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Adsorption process is another method that is cheaper and have high efficiency compare to other technologies. Factors that affected the adsorption rate are the type of adsorbent, dose of adsorbent, contact time, initial concentration and the pH of adsorption medium [63,71-73]. The common adsorbent like activated carbon have been studied in the removal of 2,4-D from the water (3). Other than activated carbon, single-walled carbon nanotubes have been used as an adsorbent due to their large surface area which have high adsorption rate [62]. In addition, adsorbent from nanoporous materials like Cr-benzenedicarboxylate, MIL-53 which is a metal-organic framework has been prove that this adsorbent is effective and has fast adsorption rate because of their high and regular porosity [72]. In order to reduce the disposal of industrial wastes, the silica gel from factory waste can be surface modified to become an adsorbent for the adsorptive removal of 2,4-D for waste water treatment [71]. Study [71] found out that the presence of electrolyte and pH solution influence the performance of surfactant modified silica gel waste (SMSGW). Biological process is different from chemical and physical process as this method use microorganism to remove 2,4-D from the water. One of the example is to use 2,4-D degrading granular sludge cultivated through bioaugmentation of glucose-fed granules with a pJP4 plasmid donor strain to degrade mixture of 2,4-D and the chlorophenols [74]. Report [74] prove that this mixture of organic pollutants can be biodegraded and mineralized by the granules as a carbon source in 420 days. Another biological process is to use a microbial community that can degrade 2,4-D as their carbon and energy sources in a continuously operated packed-bed column (PBC) and this study have high removal of 2,4-D (91.75%) within 150 days or operation [63]. Both of the experiments show that these method can improve the biomass and bioactivity of the process. The treatment process for pollutant from 2,4-D is challenging as this herbicide comes with various by-products, depending on the type of treatment process involved. Therefore, not all of the single treatment can fully remove or oxidize the organic pollutant from water. Sometimes, they need to be combined with another method to achieve the maximum rate of removal. There are a few of research use electrochemical technology as the pre-treatment process and then follow up by another process such as biological treatment or Fenton-like process [64,65].

**CONCLUSION**

2,4-D and its derivatives have been the most widely used herbicides in all over the world. Their popularities among farmers and other users are undeniable. They have gave the world a lot of benefits in many ways. Despite their sophisticated contributions, they had caused a lot of problems to the environment. These herbicides had caused contamination in various environmental bodies including water, soil and air. Due to many reasons, their distribution in the environment has become broad and they can pollute the food source. Human and other organisms are exposed to these herbicides and this situation can cause much adverse effect to their health and
growth. Many factors influence their fate and persistence in the environment. There are limited information about behavior of 2,4-D derivatives in the environment. Therefore, a lot of research must be done to study their route in the environment after their application in order to monitor and control the usage of these herbicides. The environmental fate and negative impact of these herbicides has become a great concern and it is vital to evaluate regularly. Their toxicity effects also become another serious issue as the problem occur globally. The study of their toxicity effect should be carried out more often especially on 2,4-D n-butyl ester, as there is little information about these herbicides. The study of their toxicity behavior in animal and human are really important and can be used to prevent misuse of these herbicides. In order to treat this herbicide, many methods have been established. The treatment methods have been studied and it is proven that they are efficient in their own ways. All of these studies are vital to educate people about the awareness on the pollution of these herbicides.

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