

# Electron beam decomposition of pollutant model compounds in aqueous systems

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**Abstract.** In this study, factors that limit the utilization of electron beam irradiation for decomposition of pollutants were investigated. Potassium hydrogen phthalate (KHP) was used as the model pollutant compound. The accumulated chemical oxygen demand (COD) removal below 2.4 kGy (applied for concentration at 85, 170, 255, 425 and 850 mg/l) was less than 10%, whereas the accumulated COD removal at 215 kGy (applied for concentration at 85, 170, 255, 425 and 850 mg/l) was 198%. At a lower concentration of KHP, 85 mg/l, the accumulated COD removal (for dose ranging from 0.5 to 215 kGy) was 236%. As the concentration of KHP increases to 850 mg/l, almost no COD removal was recorded (for dose ranging from 0.5 to 215 kGy). The results show that the removal efficiency and effectiveness of pollutants were influenced by the solute concentration and irradiation dose. When the concentration of KHP increases to a certain level, water radical species were not sufficient to form KHP radicals and, therefore, reduce the decomposition of KHP molecules. Removal of phthalate as measured by COD was restricted at a very high concentration, 850 mg/l. This indicates that the by-products have scavenged the radicals rapidly. This also suggests that, the limitation of radiolytic products of water to decompose KHP at higher concentration. A large quantity of KHP molecules would also stabilize the KHP radicals and resulting in lower removal of KHP. Similar removal trend was also observed for the actual industrial wastewater.

**Key words:** absorbed dose • concentration of solute • ionizing radiation • organic decomposition

## Introduction

Application of ionizing radiation for recalcitrant organic decomposition has been well documented for such compounds as methyl *tert*-butyl ether (MTBE) [3], phosphonic acid, dimethyl ester [17], 2,4-dichlorophenoxyacetic acid [6], aromatic hydrocarbons [12], benzene, toluene, *m*-xylene, *o*-xylene [16], THMs [5], bromate [21], phenol [14], methyl isobutyl ketone, toluene, xylene, tetrachloroethylene (PCE), trichloroethylene (TCE) [7, 8] and others. Studies on the decomposition of many other individual pollutants using various advanced oxidation processes can be found in the literature, e.g. a review on the decomposition of biphenols, alkylphenols and phthalates by advanced oxidation processes [10].

Industrial activities are well known of generating wastewater that lead to the environmental degradation which has become a serious problem. Wastewater generated from industry has a high complexity of pollutants which include hazardous and toxic materials. For example, in the textile industry wastewater effluent is consisted of a mixture of many individual pollutants.

Although ionizing radiation has been regarded as effective method to decompose individual pollutant by analyzing the concentration of individual pollutant, the efficiency and effectiveness of decomposition after irradiation are influenced by many factors. All these

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limiting factors can be found in the literature, such as dose, concentration of pollutants, *G*-values, etc. These limiting factors will also be discussed in this study but the primary objective of this study is to specifically evaluating the characteristic of mineralization of pollutants using ionizing radiation technique by measuring the COD. In addition to that, this study will attempt to use concentrations of phthalate from 10 mg/l to 850 mg/l. Concentration of phthalate at very high concentrations were chosen so that complete data can be obtained within reference to the limitation of phthalate removal. This will also give a detailed understanding and characterization of the destruction of phthalate as measured by COD. It is expected that, when solution has very high concentration of pollutants, the by-products will scavenge the radicals rapidly. Hence, this study is not aiming to employ free radicals from irradiation to destruct the pollutants at a concentration as high as 850 mg/l of phthalate.

Decomposition of individual pollutant by measuring its concentration left after irradiation treatment is different comparing to the measured COD which is monitoring the degree of complete mineralization of complex or aggregate pollutants after irradiation treatment. The parameter of COD has been used to monitor the general status of wastewater effluent and quality of treated water. It is not practical to identify or separate individual pollutant found in wastewater effluents.

Chemical oxygen demand is widely accepted by the regulatory body to monitor the discharge of treated effluent and it must comply with the standard. It is used to indicate the aggregate amount of organic matters present in wastewater and determine the quality of water [1]. The measurement of COD is based upon the theoretical amount of oxygen required to oxidize organic compounds to CO<sub>2</sub> and H<sub>2</sub>O.

Research works on the application of ionizing radiation for COD removal have also been carried out in combination with conventional processes such as physical, chemical and biological process [2, 7, 11, 18–20]. There are other works studying the effectiveness of irradiation to reduce or remove COD and some results did not indicate reduction of COD after irradiation [7]. This study, therefore, attempts to characterize the effectiveness of ionizing radiation in removing/reducing COD. Analyzing the COD, indicates the degree of mineralization of irradiated sample in aggregate conditions. Therefore, it is relatively different compared with the analysis of the concentration of individual targeted pollutant of irradiated sample. In this study, an individual compound is subjected to treatment by using ionizing radiation. Instead of measuring the efficiency of individual pollutant decomposition through determination of concentration before and after radiation treatment, this study focuses on measuring the COD of individual pollutants.

By measuring the COD of individual compound and real wastewater, this study is aimed to investigate the effectiveness and limitation of ionizing radiation in mineralization of irradiated sample. This study also compares the mineralization of individual pollutant, namely phthalate with real wastewater from different industries such as the food and beverages industry and the textile industry. Perhaps it is difficult to extrapolate directly the result of mineralization of individual pollutant with real wastewater, however, the trend of mineralization between individual pollutants and real wastewater obtained may give further understanding on the characterization of irradiated sample as measured by COD.

Potassium hydrogen phthalate was applied as individual pollutant and was selected because it is the most commonly used compounds for calibration of COD [1].

## Experimental

### Sample preparation and irradiation

Artificial wastewater sample was prepared using KHP obtained from Merck, Germany. The concentrations of phthalate at 10, 40, 85, 170, 255, 425 and 850 mg/l in aqueous solutions were prepared using distilled water. Meanwhile, textile and food and beverages wastewater were collected from the Integrated Industrial Park located in Selangor, Malaysia. The characteristic of the wastewater is shown in Table 1.

Samples were placed on the trolley and delivered to the irradiation chamber for irradiation. Irradiation of the sample was performed using an NHV (Nissin-High Voltage), electron accelerator, EPS 3000 at Nuclear Malaysia. Samples were irradiated at actual pH without adjustment. The energy of 1 MeV of the electron accelerator was selected for all of the irradiated samples, their thickness of between 3–4 mm. The speed of the conveyor and current (mA) of the electron accelerator were adjusted according to dosages required. Dosimetry of the sample was conducted using a CTA film (FTR-125, Fuji Photo Film Co.). The doses selected for this study were: 0.5, 2.4, 8, 18, 41, 53, 108 and 215 kGy.

### Sample analysis

Analyses of samples were conducted in accordance with APHA *et al.* [1]. COD was determined by using dichromate solution with digestion in a Hach reactor and followed by UV absorption measurement using a Hach-2400 spectrophotometer. pH of the water sample was measured using a pH meter, WTW Multi

**Table 1.** Characteristics of samples from different types of wastewater

Parameter	KHP	Textile	Food and beverages
pH	4.10	10.68	4.15
COD (mg/l)	1000	960	3080
TSS (mg/l)	< 1	1246	380
BOD (mg/l)	50	146	1294
Turbidity (NTU)	< 1	10	208

340i. Turbidity was determined using a Hach 2100 P turbidimeter. BOD (biochemical oxygen demand) was measured using a Lovibond OxiDirect meter. Total suspended solid (TSS) was measured using a glass microfibre filter (GF/B). Shimadzu high performance liquid chromatography (HPLC) – SPD6AV equipped with Phenomenex (Gemini 5  $\mu\text{m}$ , C18, 110 A) was used for the determination of phthalate ions with the UV Shimadzu spectrophotometer at 280 nm and employing an eluent consisting of 70%:30%, acetonitrile (Fisher, UK):H<sub>2</sub>O.

### G-value

The *G*-value defined as the number of molecules decomposed per 100 eV of the absorbed dose was calculated using Eq. (1), [3]:

$$(1) \quad G = [R] \times (6.023 \times 10^{23}) / D \times (6.24 \times 10^{16})$$

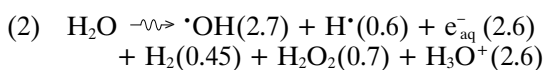
where: *[R]* is the change of concentration ( $\text{mol}\cdot\text{l}^{-1}$ ) of solute at given dose; *D* is the absorbed dose (Gy);  $6.023 \times 10^{23}$  is Avogadro's number;  $6.24 \times 10^{16}$  is the conversion factor from Gy to 100 eV/L.

## Results and discussions

### Effects of irradiation dose and concentration of solute

Figure 1 shows the effect of irradiation doses on the accumulated percentage removal of COD for different concentration of KHP (the accumulated COD removal (%) used in Fig. 1 is derived by adding the removal of COD, all initial phthalate concentrations irradiated at a given dose). The rate of COD removal increases with increasing irradiation doses. The accumulated COD removal at 0.5 and 2.4 kGy were below 10%, whereas the accumulated COD removal at 215 kGy was 198%.

Ionizing radiation of water will lead to the formation of free radicals [23] as follows:



The yield of species per 100 eV of the absorbed dose of *G*-value is given by the number in parentheses. Hydroxyl radical is the main reactive species respon-

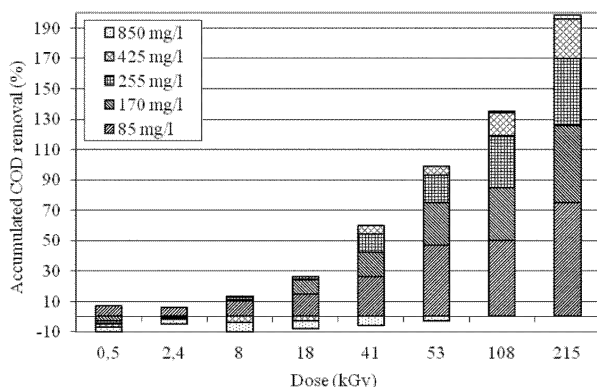


Fig. 1. Effect of absorbed dose in COD removal.

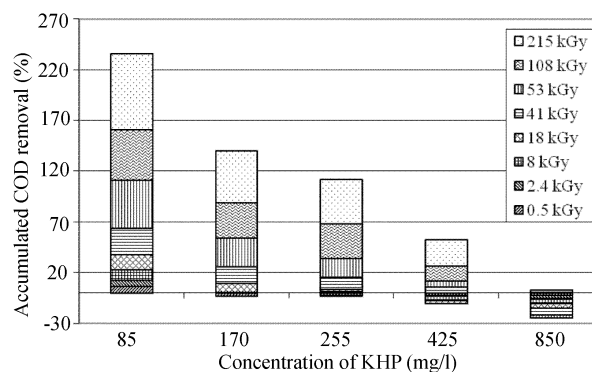


Fig. 2. Effect of the concentration of solute in COD removal.

sible for the decomposition of pollutants. Hydroxyl radical produced will attack the aromatic molecules and produce intermediates through two pathways: H atom abstraction and addition to aromatic rings [9, 13]. These intermediates will lead to the mineralization of the pollutants. It has been shown that OH radicals can attach to the benzene ring of phthalate at the following rate,  $k = 5.9 \times 10^9 \text{ L}\cdot\text{mol}^{-1}\cdot\text{s}^{-1}$  [22] which will lead to ring opening and degradation of the phthalate. Similar effect is expected when KHP is exposed to OH radicals. The higher the dose, more OH radicals are produced and hence increase the degradation of phthalate.

However, the COD removal is also influenced by the concentrations of solute (Fig. 2). The accumulated COD removal (%) used in Fig. 2 is derived by adding the removal of COD irradiated from 0.5 to 215 kGy at a given initial concentration of phthalate. The accumulated COD removal for concentration of solute at 85 mg/l was 236%. As the concentrations of solute increase, removal of COD was reduced. For the concentration of solute at 850 mg/l, almost no COD reduction was recorded for most of the doses applied (Fig. 2). Not only the value of COD (%) did not reduce after irradiation at concentration of solute at 850 mg/l, there was a slight increase of COD (value-mg/l) after irradiation. The increased COD (value-mg/l) after irradiation was indicated by the negative value of COD (%) (Fig. 2). This suggests that at higher concentration of pollutants, they were remained in the incomplete decomposition condition. The breakdown of the pollutants to smaller or simpler form after irradiation may result into a slight increase of COD value (mg/l). No reduction of COD value (%) after irradiation also indicates that almost no mineralization of phthalate (to CO<sub>2</sub> and H<sub>2</sub>O) was recorded after irradiation. High concentration of phthalate at 850 mg/l, has resulted in restriction of destruction of phthalate by radicals and this was likely due to the by-products scavenge most of the free radicals introduced to the aqueous systems.

The factors affecting the removal of pollutant were further characterized and shown in Figs. 3 and 4. The removal of KHP for the concentration between 10 to 255 mg/l was measured by UV absorption at 280 nm at an irradiation dose from 8 to 215 kGy. There was no reduction of KHP for the concentration above 425 mg/l at the irradiation dose given above. On the other hand, at low concentration of KHP, there was no reduction of KHP recorded for the absorbed dose of 2.4 kGy and below. The above results indicate the limitation of radiation

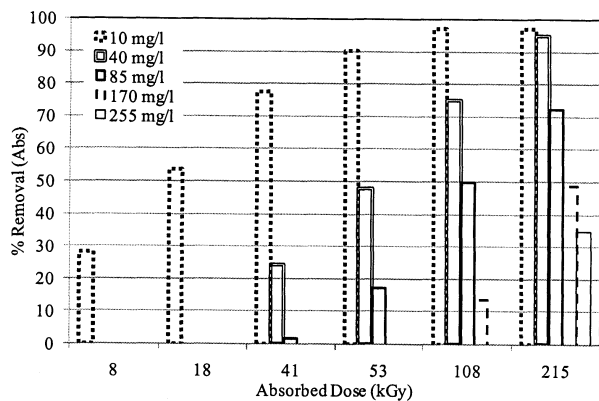


Fig. 3. Removal of KHP (%) after irradiation.

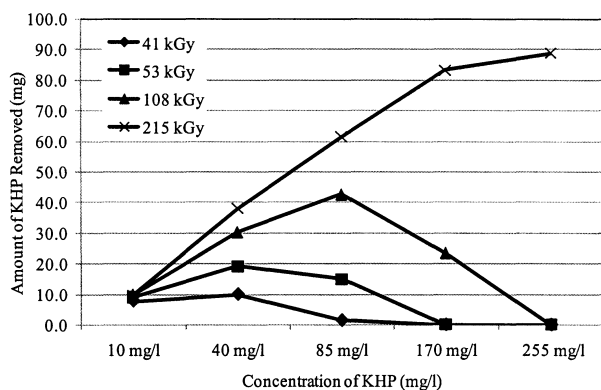


Fig. 4. Quantity of KHP (mg) removed after irradiation.

degradation of organic pollutants by the irradiation dose and the concentration of the solutes. As the concentration of KHP increases, higher dose was required to obtain first removal of KHP (Fig. 3).

When the concentration of the initial KHP increases from 10 to 255 mg/l, the percentage removal of KHP decreases for irradiation doses from 41 to 215 kGy (Fig. 3). Although there was a decrease in percentage of the removal when the initial concentration of KHP increased from 10 to 85 mg/l, the quantity (mg) of KHP removal did not necessarily reduce (Table 2).

At a lower irradiation dose of 41 kGy, the amount of KHP removed decreases from 7.7 to 1.4 mg/l when the initial concentration of KHP increases from 10 to 85 mg/l. This indicates that, at the dose of 41 kGy, the increase of initial concentration of KHP from 10 to 85 mg/l has limited the removal of KHP. When the dose remains the same (at lower range of dose), the increases of the concentration of KHP has reduced the quantity removal of KHP. At low concentration of KHP, the amount of water radical species generated was sufficient to react with 77% of the KHP molecules to form KHP radicals and subsequently degraded into smaller compounds. At higher concentration of KHP, water radical species were not sufficient to react with a larger amount of KHP to form KHP radicals and hence reduce in the degradation of KHP molecules. Large amount of KHP molecules also may stabilize the KHP radicals and hence further reduce the % removal of KHP.

For doses of 53, 108, and 215 kGy, the amount of KHP removed has increased from 9 to 14.8 mg/l, 9.7 to 42.5 mg/l and 9.7 to 61.4 mg/l when the initial concentration of KHP increased from 10 to 85 mg/l (Fig. 4 and

Table 2. Removal of KHP in percentage (%) and quantity (mg) after irradiation

Dose (kGy)	for 10 mg/l		for 40 mg/l		for 85 mg/l		for 170 mg/l		for 255 mg/l	
	% of mg/l removed	Quantity (mg) removed	% of mg/l removed	Quantity (mg) removed	% of mg/l removed	Quantity (mg) removed	% of mg/l removed	Quantity (mg) removed	% of mg/l removed	Quantity (mg) removed
41	77.46	7.7	24.44	9.8	1.61	1.4	0.00	0.0	0.00	0.0
53	90.14	9.0	47.78	19.1	17.38	14.8	0.00	0.0	0.00	0.0
108	97.18	9.7	75.19	30.1	50.00	42.5	13.71	23.3	0.00	0.0
215	97.18	9.7	94.81	37.9	72.22	61.4	48.87	83.1	34.76	88.6

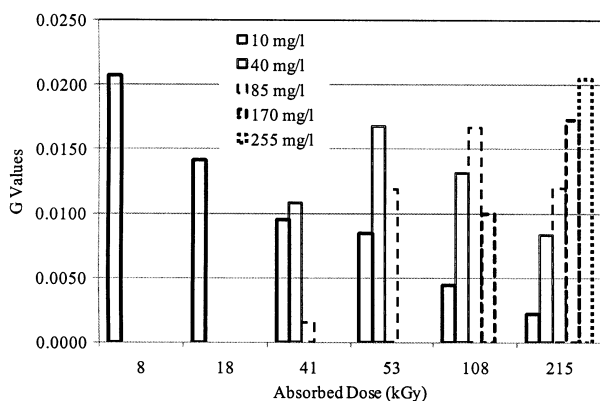
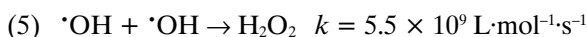
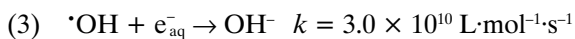


Fig. 5. *G*-values of KHP at various doses.

Table 2). This shows that at a given absorbed dose, the percentage removal of KHP has reduced when higher initial concentration of KHP was used. But the quantity of KHP (mg) removed has increased vs. concentration at a given absorbed dose.

When the initial concentrations of solute increase to 850 mg/l, no removal of KHP was recorded for the dose of up to 215 kGy. This indicates that when high dose was introduced, very high concentration of solute in the aqueous system would restrict the interaction of electron from electron beam irradiation with the water molecules. This may due to the stabilization of KHP radicals resulting from large amounts of KHP molecules. High concentration of phthalate at 850 mg/l also has led to the scavenging of the radicals rapidly by the by-products.

Figure 5 shows the *G*-values obtained based on the data from Fig. 3. For a given concentration, the removal efficiency as reflected with *G*-value has displayed two trends. The first trend, at a solute concentration of 10 mg/l, where the *G*-values have shown a decrease versus absorbed dose. This trend was also observed in other studies [3, 14, 24] where lower concentration of solute was employed. This suggests that, for a given concentration, higher dose would result in higher reactive radicals formed, but leads to the increase of radicals-radicals recombination involving  $\cdot\text{OH}$ ,  $e_{\text{aq}}^-$  and  $\text{H}\cdot$  (Eqs. (3), (4) and (5); [4]).



As the dose increases, the competition of radicals reaction in radicals-radicals recombination reactions and radicals-solute reactions have resulted in lower removal efficiencies.

Second trend was observed for the concentration of solute at 40 mg/l and above, where *G*-value has increased vs. absorbed dose, and subsequently decreases vs. absorbed dose (Fig. 5). To the best knowledge of the present authors this trend has not been reported before because most of the studies related to *G*-value were working at a lower range of pollutants concentration. The result obtains from this study (at a concentration of 40 mg/l and above) indicates that when the initial

concentration of solute was high, the removal efficiency, as shown by the *G*-value, would increase vs. absorbed dose. Subsequently, this was followed by the reduction of *G*-value. This suggests that, for a given initial concentration of solute (for concentration 40 mg/l and above), radicals-solute reactions have a higher yield compare to the radicals-radicals recombination as the absorbed dose applied increases. After it has approached the limit vs. absorbed dose, the higher dose would lead to higher radicals-radicals recombination compare to radicals-solute reactions.

#### Degradation of KHP: HPLC analysis

Result obtained from HPLC analysis was in agreement with removal of the concentration of KHP as described earlier. The HPLC chromatogram of solute with a concentration of 85 mg/l was illustrated in Fig. 6. The highest removal of KHP was recorded at 215 kGy. As the concentration of KHP increases to 850 mg/l, the HPLC chromatogram did not show reduction for all the doses of up to 215 kGy. HPLC chromatogram is expected to show signal of by-products of decomposition. But surprisingly, we did not obtain the signals of

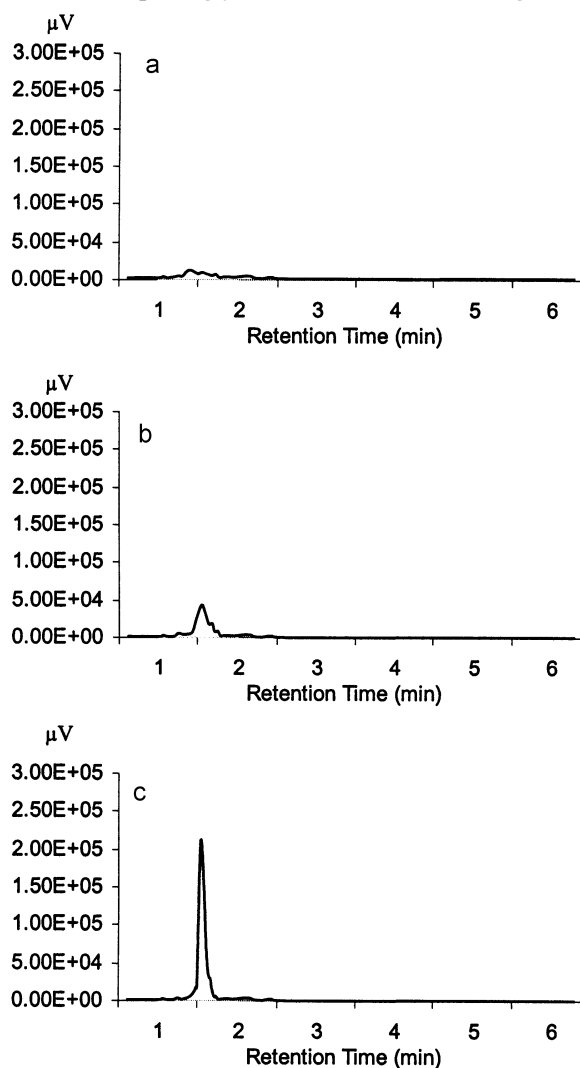
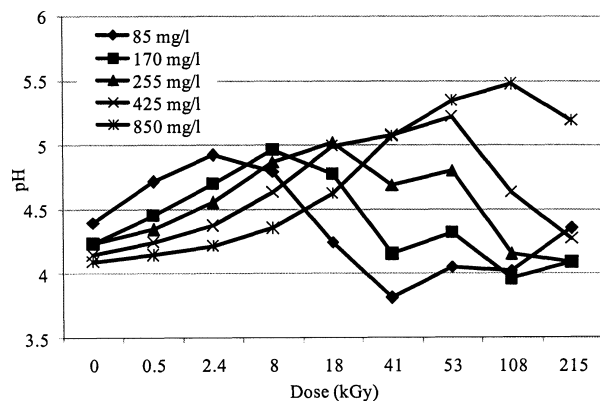


Fig. 6. Decomposition of KHP with initial concentration of 85 mg/l at 215 kGy (a); 41 kGy (b) and 0 kGy (c).



**Fig. 7.** The effects of absorbed dose and concentration of solute on the pH.

by-products of decomposition even after retention time of above 30 min.

#### Effects of the absorbed dose and concentration of solute on pH

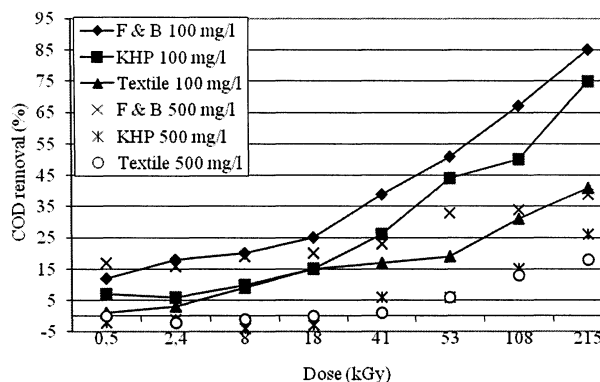
For non-irradiated samples of KHP, their pH values decrease when the concentration of KHP increases (Fig. 7). The decreased pH values could be attributed to a higher portion of water when the concentrations of KHP is reduced. The effect of irradiation on the pH was influenced by the absorbed dose and the concentration of solute. The pH of the samples irradiated at a dose of 8 kGy and below has increased vs. dose. However, for samples irradiated at a dose of 8 kGy and above, the pH values have started to decrease vs. dose. The pH of the lowest concentration of KHP started to decrease first, and subsequently followed the higher concentration of KHP vs. dose (Fig. 7). The changes of pH values after radiation treatment is expected to be contributed by the breakdown of phthalate compound. However, we are unable to explain the two separate trends (increase and reduce) of each concentration vs. adsorbed dose.

#### Comparison with different types of wastewater

COD removal of textile and food and beverages wastewater by ionizing radiation have shown a similar trend with KHP. Both concentration of pollutants and irradiation dose have influenced the decomposition of pollutants at different magnitude. Initial concentration of pollutants with COD value of 100 mg/l has recorded higher removal percentage compared to 500 mg/l (Fig. 8).

Type of wastewater may also determine the degree of COD removal with the following order, F and B > KHP > textile. Total suspended solid (TSS) for KHP, food and beverages wastewater and textile wastewater was < 1, 380 and 1246 mg/l, respectively. The biodegradability index for KHP, textile and food and beverages was 0.05, 0.15 and 0.42, respectively.

The food and beverages industry wastewater has a relatively low molecular weight with a simple form of organic compounds. Lower molecular weight and simple form of the organic content will lead to a higher



**Fig. 8.** Effect of the type of wastewater on COD removal by ionizing radiation.

biodegradability index. Wastewater that comprises larger molecular weight and refractory organic compounds will result in a lower biodegradability index. Wastewater is considered easily biodegradable if it has a biodegradability index > 0.3–0.4 [15]. This may lead to a higher percentage of COD removal for the food and beverages industry compares to other wastewaters. This suggests that the decomposition of food and beverages industry wastewater is easier compared to KHP and textile wastewater that have a higher molecular weight and complex refractory organic compounds.

KHP contains one compound and the composition of real wastewater is very complex and unknown. The direct comparison of COD removal for KHP perhaps is difficult to extrapolate with real wastewater. However, the results indicate that all types of wastewater have displayed a similar trend of pollutants removal utilizing electron beam irradiation. Thus, the relationship between limiting factors, characteristics and mechanism involved can be related when utilizing ionizing radiation technology for decomposition of pollutants.

#### Conclusions

Ionizing radiation is an effective method to decompose individual recalcitrant organic pollutants. However, pollutants present in industrial wastewater are complex and comprise a substantial number of unknown compounds. Although the degree of complexity of pollutants found in wastewater will influence the degree of decomposition, the concentration of pollutants found in the wastewater is more significant in influencing the degree of decomposition. When the concentration of pollutant is too high, water radical species will likely become insufficient to react with pollutants. Besides, by-products found in the aqueous system also would scavenge the radicals rapidly. This will lead to less or limited reaction between radical species and pollutants for decomposition. By introducing the right absorbed dose and concentration, ionizing radiation is an effective method to decompose aggregate recalcitrant organic pollutants, as measured by COD, into partial and complete decomposition of recalcitrant organic compounds. Based on the result obtained in this study, the application of ionizing radiation alone may be not suitable to treat wastewater with concentration of COD above

100 mg/l. Treating wastewater using ionizing radiation alone with concentration of COD above 100 mg/l not only require high adsorbed dose (high cost), it also is unable to achieve high rate of mineralization. This is because at very high concentration of pollutants, the by-products will scavenge the radicals. Therefore, free radical destruction in high concentration of pollutants is not likely to take place. The concentration of initial COD above 100 mg/l may require a combined approach such as ionizing radiation and biological system.

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