



## Original Article

# Evaluation of decontamination factor of radioactive methyl iodide on activated carbons at high humid conditions



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## ABSTRACT

Radioactive iodine ( $^{131}\text{I}$ ) released from nuclear power plants has been a critical environmental concern for workers. The effective trapping of radioactive iodine isotopes from the off-gas stream generated from nuclear facilities is an important issue in radioactive waste treatment systems evaluation. Numerous studies on retaining methyl iodide ( $\text{CH}_3^{131}\text{I}$ ) by impregnated activated carbons under the high content of moisture have been extensively studied so far. But there have been no good results on how to remove methyl iodide at high humid conditions up to now. A new challenge is to introduce other promising impregnating chemical agents that are able to uptake enough radioactive methyl iodide under high humid conditions. In order to develop a good removal efficiency to control radioiodine gas generated from a high humid process, activated carbons (ACs) impregnated with triethylene diamine (TEDA) and quinuclidine (QUID) were prepared. In addition, the removal efficiencies of the activated carbons (ACs) under humid conditions up to 95% RH were evaluated by applying the standard method specified in ASTM-D3808. Quinuclidine impregnated activated carbon showed a much higher decontamination factor above 1,000, which is enough to meet the regulation index for the iodine filters in nuclear power plants (NPPs).

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## 1. Introduction

Radioactive isotopes of iodine are potentially hazardous fission products that could be generated as a uranium fission product during the nuclear power plants' (NPPs) operations, as they can critically impact the workers and the environment. Among the various types of isotopes, iodine-131 ( $^{131}\text{I}$ ) released as gaseous effluents from the nuclear power plants (NPPs) has been a critical environmental concern. This is due to the combination of its large inventory in irradiated nuclear fuel, a half-life of 8.05 d for the isotope of greatest radiological significance ( $^{131}\text{I}$ ), a complex chemistry that may lead to the formation of volatile species, and its hazardous biological effects on the thyroid. Removing these radioactive iodine isotopes from the gaseous effluents of NPPs is an important issue in radioactive waste treatment system evaluation, especially in their adherence to the “as low as reasonably achievable” (ALARA) criteria [1,26].

Under normal power operating conditions, the radioactive iodine release rates from the core and reactor coolant are very low.

In one study, for example, airborne iodine  $^{131}\text{I}$  was measured in the radioactive wastes treatment system of a German pressurized water reactor (PWR) [1]. Concentrations below  $400 \text{ Bq/m}^3$  were detected at the inlets of the exhaust air filters for the reactor containment systems. Typical annual discharge rates for European reactor stations are in the range of  $10^7$ – $10^9 \text{ Bq/yr}$  of iodine-131 [1]. An average  $^{131}\text{I}$  discharge of  $5.6 \times 10^9 \text{ Bq/yr}$  is usual for pressurized water reactors (PWR) in the United States [2].

In accidental situations, the amounts of iodine released into the containment systems and the environments depend on many factors. The plant's design construction details, and the sequence of the events leading to the accident, are very important. In the case of severe accidents, however, the actual quantities released may represent a significant percentage of the core inventory. Thus, estimates for individual nuclear power plants indicate their potentials [3–6]. Radioactive isotopes of iodine generated from nuclear power plants (NPPs) are partially converted radioactive organic iodide, such as methyl iodide ( $\text{CH}_3^{131}\text{I}$ ), which react with organic matter in the process line. Because radioiodine is one of these gaseous wastes that can cause radiological harm on the human body and environment, it is important to remove this gas below the restriction of release, even in the case of normal operating and

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accident conditions. Therefore, numerous studies for the removal of radioiodine from the off-gas stream of nuclear facilities have been performed with the formation processes and chemical structure [1–6,26].

The removal of the radioactive isotopes from iodine have generally been performed using activated carbon (AC), silica, alumina, and polymer resin [1–3,5,6,26]. Also, impregnants such as KI, TEDA (triethylene diamine), and QUID (quinuclidine) have been widely used to improve the removal efficiency. The activated carbon is a widely used adsorbent for trapping radioactive iodine gases to protect personnel and environments from various nuclear facilities, including nuclear power plants (NPPs), and it also applicable for military respirators and purification apparatus from various toxic gases vapor and liquid chemicals because it has high porosity and hydrophobicity [7–10]. The impregnated TEDA on activated carbons can convert to quaternary ammonium salts by reacting with organic iodides as follows:

There has been some concern regarding the use of TEDA-impregnated activated carbon (AC) as an adsorbent for radioiodine removal because of pore blocking and the possible release of TEDA from the charcoal in continuously moist air streams. Therefore, the TEDA-impregnated activated carbon showed a rapid drop in the removal efficiency of activated carbon (AC) to trap methyl iodide ( $\text{CH}_3\text{I}$ ) generated from high humid conditions [11–13,26] (see Fig. 1).

Many researchers have tried to enhance the radioactive iodine trapping efficiency by modifying the surface functional groups and porosity of activated carbon or by impregnating with other chemicals [9–16]. Due to its many advantages including a low production cost, easy to control the surface hydrophobicity, and wide applications, activated carbon (AC) has been a widely used adsorbent for removing radioactive iodine isotopes to protect personnel and environments [7]. To enhance the adsorptive removal performance, the surface functional groups and porosity of AC have often been modified or impregnated with other chemicals [8–11,26]. Nowadays, quinuclidine (QUID) has been introduced as a prominent impregnating chemical for removing the radioactive isotopes of iodine. The molecular weight of a QUID (111.19 g/mol) is similar to that of a TEDA (112.18 g/mol), but the basicity of the former ( $\text{pK}_a = 11.3$ ) is much higher (TEDA:  $\text{pK}_a = 8.7$ ) [7,8]. QUID contains an amine group at one end of the molecule while the opposite end of the molecule permits its ready adsorption and retention on the preferred adsorbent activated carbon, as shown in Fig. 2.

Many previous studies were carried out to evaluate the removal efficiencies of TEDA-AC by changing various test conditions such as bed temperature, bed depth, and amount of impregnant. In the research on the novel impregnants for activated carbons to enhance their chemical activity, we prepared two activated carbons impregnated with TEDA and QUID and experimentally evaluated the decontamination factors of the ACs under high contents of moisture condition up to 95% RH by applying ASTM-D3808 [12,13].

## 2. Experimental and material

### 2.1. Preparation of materials

The two types of activated carbons (ACs) impregnated with TEDA and QUID were prepared to evaluate the decontamination factor (DF) of methyl iodide ( $\text{CH}_3^{131}\text{I}$ ) under a highly moisturizing condition up to 95 RH%. The base material used in this experimental evaluation was activated carbon. Activated carbons were prepared by impregnating two different chemical reagents of TEDA and QUID, respectively. Each adsorbent was immersed in the solution for 60 min and dried at 90 °C for 9 h in the oven. The amount of impregnants on ACs were obtained by the extraction of TEDA and QUID with acetonitrile solution and then the analysis of solution

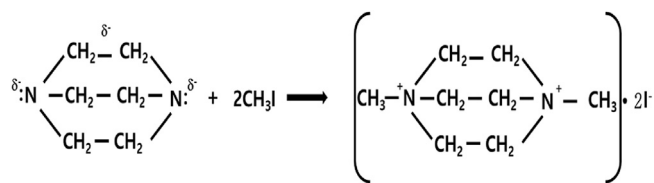


Fig. 1. Reaction mechanism of radioactive methyl iodide with TEDA on activated carbons.

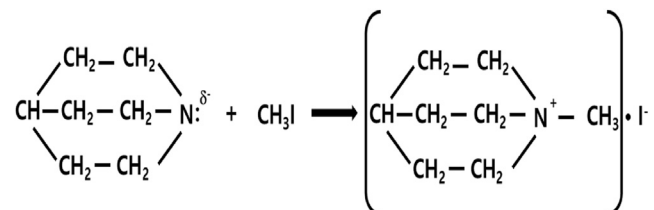


Fig. 2. Reaction mechanism of radioactive methyl iodide with QUID on activated carbons.

concentration using UV-Spectrophotometer. The physical property of two carbons with the aid of the BET-N2 (Micromeritics, ASAP 2400) analysis was obtained, as listed in Table 1. The physical properties of TEDA-AC and QUID-AC were similar to these of the AC, except for a slight decrease in the pore volume and surface area.

### 2.2. Test facility and conditions using radioactive methyl iodide ( $\text{CH}_3^{131}\text{I}$ ) tracer

The evaluation test of radioactive methyl iodide at a high moisture condition was conducted by the standard test method, as established in ASTM D3803-91 [14]. This standard covers the test method for establishing the capability of new and used activated carbon in removing gaseous radioactive iodine and iodine compounds from air and gas streams. This test determines the penetration of only  $^{131}\text{I}$ -labeled ( $\text{CH}_3^{131}\text{I}$ ) through TEDA or QUID impregnated activated carbon under a normal contaminated operating condition (30 °C, 95% relative humidity). A schematic diagram of the test facility for this research is shown in Fig. 3. Based on this schematic diagram, we designed and constructed a test facility that included related components with required functions. The individual components of this facility consist of four systems, such as a gas supply system and humidity control section, test and backup bed assemblies, and radioactive iodine ( $\text{CH}_3^{131}\text{I}$ ) supply section. Bed canisters were constructed in accordance with ASTM D-3803-91 in all dimensions. The test bed canister was a single unit of the depth specified in the test and assembled from separate 50 mm deep canisters. A backup bed canister was also installed to trap completely radioactive methyl iodide that penetrated through a sample canister. Operating conditions for the test are listed in Table 2. The test adsorbent was packed in a stainless-steel canister assembled with clamps. The total depth of the assembled two canisters was 100 mm, and two canisters were packed with an

Table 1  
Physical characteristics of Adsorbents.

Characteristic	TEDA-AC (6.5 wt%)	QUID-AC (6.2 wt%)
Mean Pore Diameter(Å)	13.2	13.4
Pore Volume( $\text{cm}^3/\text{g}$ )	0.658	0.635
Micro Pore Volume( $\text{cm}^3/\text{g}$ )	0.523	0.515
Surface area( $\text{m}^2/\text{g}$ )	1275	1265

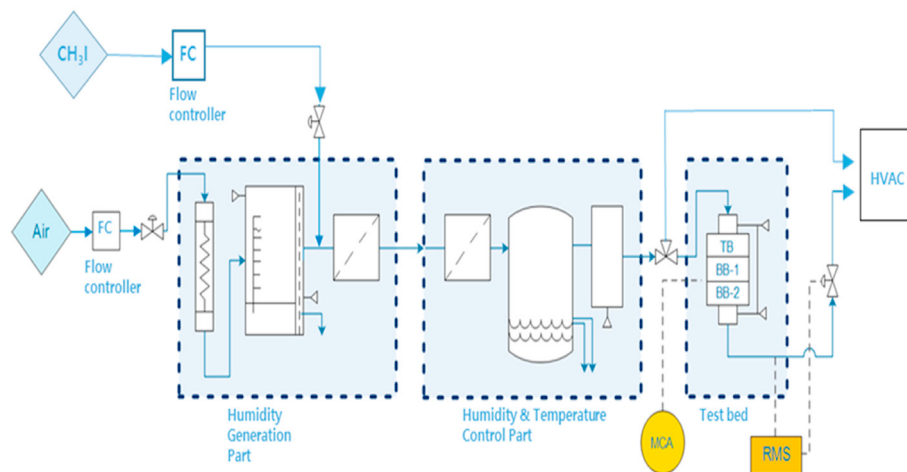


Fig. 3. Schematic diagram of test facility for removing radioactive methyl iodide.

Table 2

Test conditions for removing CH<sub>3</sub><sup>131</sup>I established in ASTM D3803-91.

Parameters	Pre-equilibrium period (First 16 h)	Equilibrium, Challenge and Elution periods (Final 4 h)
Temperature, °C	30 ± 0.5	30 ± 0.5
Relative humidity, %	92–96	93–95
Flow rate, l/min	10–25	10–25
Linear velocity, m/min	12.2	12.2
Absolute pressure, kPa	101 ± 5	101 ± 5
Adsorbate concentration, mg/m <sup>3</sup>		1.75 ± 0.25
Test duration		
Pre-equilibration, hr	16 ± 0.1	
Equilibration, min		120 ± 1
Challenge, min		60 ± 1
Elution, min		60 ± 1

adsorbent of about 56.8 g per canister for each run.

The test facility was equipped with a compressed air supply and humidity control system. All of the flow lines were equipped with check valves to prevent the back flow. The flow rate of air was controlled by a mass flow controller (MFC). The water temperature maintained a constant temperature of 30 °C, and all the gas was supplied into a water bath for humidifying. Radioactive methyl iodide (CH<sub>3</sub><sup>131</sup>I) with 3.7 × 10<sup>2</sup> Bq/m<sup>3</sup> was prepared by a synthesis reaction of Na<sup>131</sup>I source with organic compounds. The reaction to synthesize radioactive methyl iodide was proceeded with isotopic substitution as follows: CH<sup>127</sup>I + 2Na<sup>131</sup>I · CH<sub>3</sub><sup>131</sup>I + Na<sup>131</sup>I ⇌ Na<sup>127</sup>I. The Na<sup>131</sup>I source was purchased from KAERI. A multichannel analyzer (MCA, Canberra GC3018) with a lead-shielded NaI detector was used for the <sup>131</sup>I detection inside a uniform shape sampler.

The decontamination factor (DF) was obtained from the penetration values (P) as follows:

$$DF = 1/[1-P/100] P(\%) = A/(A+B) \times 100$$

A: Amount of gamma activity determined to have passed the backup bed (gamma activity in backup bed, Bq)

B: Amount of gamma activity trapped in the test bed (gamma activity in test bed, Bq).

### 3. Evaluation of decontamination factor (DF) of methyl iodide

#### 3.1. Trapping efficiency of methyl iodide by TEDA-AC

In general, the amount of TEDA in activated carbon installed in a

nuclear facility has been applied at ranges from 2 wt% to 5wt. Several studies already conducted the removal efficiencies for radioactive methyl iodide by various commercial TEDA-ACs to determine the optimum amount of TEDA impregnated on ACs [17–20]. González et al. reported that 5.0 wt% impregnated TEDA-AC showed the highest removal efficiencies when it was evaluated by the standard method of ASTM D 3803 [19]. In this study, we measured the decontamination factors (DF) of radioactive methyl iodide on 6.5 wt% TEDA-AC at 95% RH by using the test facility shown in Fig. 3. All of the tests were performed according to ASTM 3803 listed in Table 2. The DFs of radioactive methyl iodide on TEDA-AC were evaluated from 502 to 683 (99.8%–99.82% in removal efficiency), as shown in Fig. 4. From the regulation index for the iodine filters in nuclear power plants (NPPs), the required removal efficiency of the iodine adsorber should be greater than 99.9% (DF ≥ 1000). The DFs of 6.5 wt% TEDA-AC measured from this study were not enough to meet the requirement of the TEDA-AC for removing radioactive methyl iodide. Previous studies already found that the removal of methyl iodide from various charcoal filters declined exponentially with an increase in the relative humidity (RH) [10–12,16–19]. Recently Keon Ho et al. reported the effect of moisture on the adsorption amounts of methyl iodide at various moisture contents up to 90% RH on tertiary amine impregnated ACs [20]. They reported that the removal efficiency of methyl iodide on TEDA-AC showed a rapid drop of 3 times compared to the dry condition as the moisture of feed stream increased at a range of 30–90% RH. As the humidity increased, the lots of water molecules inside the pores could block the active sites in pores, retard the reaction rate with methyl iodide, and finally decrease the removal

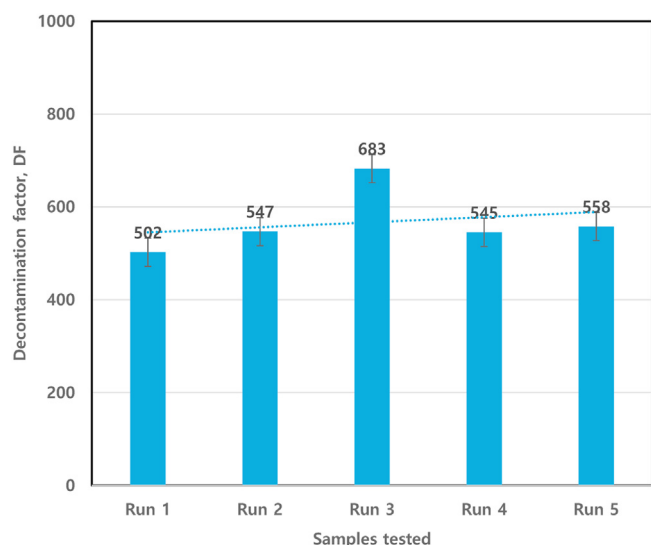


Fig. 4. Decontamination factors (DF) on TEDA-AC by ASTM D 3803. (30 °C, 95 RH, 12. 2 m/min, upstream activity 1279 Bq).

capacity, unlike the case under a dry condition. It has been also generally recognized that TEDA clearly contributed to enhancing the adsorption affinity due to the strong bond strength by chemical reaction between TEDA and water molecules in pores. It implies that the TEDA that reacted with methyl iodide in pores played a large role, and the chemical reaction with methyl iodide is more important than the physical properties (i.e., surface area, pore volume, etc.) of AC as the moisture in the feed stream increased. Removal efficiency on the TEDA-AC depend on the moisture contents of feed stream. DFs were also measured by using radioactive isotopes with high degree of safety. It seemed that data deviation are caused by a small change of moisture contents (95% RH) of feed stream.

### 3.2. Trapping efficiency of methyl iodide by QUID-AC

To evaluate the retention capability of radioactive methyl iodide on QUID-AC, decontamination factors (DF) were tested at a high humid condition of 95% RH. Fig. 5 shows that the DF of radioactive

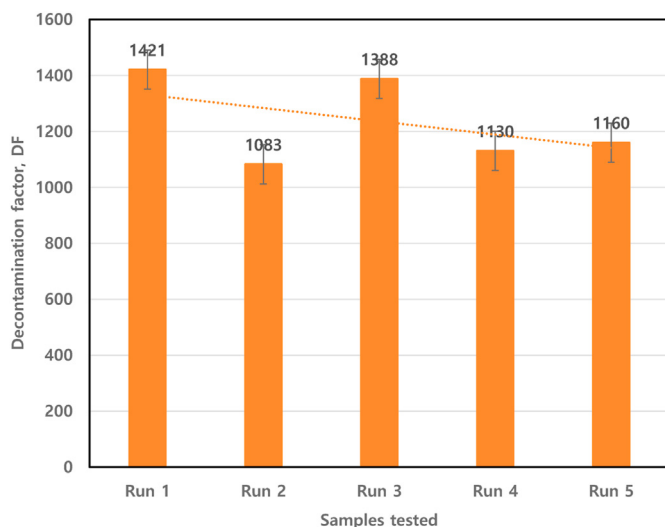


Fig. 5. Decontamination factors (DF) on QUID-AC by ASTM D 3803. (30 °C, 95 RH, 12. 2 m/min, upstream activity 1564 Bq).

methyl iodide on QUID-AC was evaluated as being more than 1000 (DF  $\geq$  1000) for the test conditions. In the case of the QUID-AC test results, the removal efficiencies of radioactive methyl iodide were evaluated to be more than 99.9%, which were enough to meet the regulation index for the iodine filters in nuclear power plants (NPPs). Compared with TEDA-AC, the DF of methyl iodide on QUID-AC had higher values than those of TEDA-AC. These results imply that the chemical reaction of QUID with methyl iodide ( $\text{CH}_3\text{I}$ ) might be more enhanced than that of TEDA because QUID formed products with a higher nucleophilicity ( $\text{Nu} = 20.5$ ) than that of TEDA ( $\text{Nu} = 18.8$ ) under a humid condition of up to 95% RH. Recently, the role of tertiary amines, such as TEDA and QUID, on the carbon surface in methyl iodide adsorption was studied by the experiments and DFT (density functional theory) calculations [21,22]. The atomic level removal process of methyl iodide molecules for TEDA-AC surfaces was investigated to identify fundamental mechanisms for the rate determining step as a function of adsorption sites, porosity, and humidity. From the simulation, it was found that the decomposition of methyl iodide into methyl and iodide ions occurs more easily in the presence of both tertiary amines and water. It was presumed that the alkylation of the tertiary amines was the major factor behind the improved  $\text{CH}_3\text{I}$  removal efficiency of the amine-impregnated AC [23,24].

Among these two different tertiary amines, QUID showed a higher trapping capability of radioactive methyl iodide at a high moisture feed stream condition up to 95% RH. These results indicate that even a small amount of the impregnant contributed strongly to the chemical reaction under humid conditions.  $\text{CH}_3\text{I}$  molecules in the feed stream were thought to be adsorbed mainly by chemical reactions under humid conditions because of the 4–5-fold reduction of their trapping capacity in the high moisture conditions relative to dry conditions [20].

Under humid conditions, the diffusion of  $\text{CH}_3\text{I}$  through the pores may be obstructed by pre-adsorbed water molecules. Therefore, it is expected that a certain amount of the impregnants moved into other pore sites when  $\text{CH}_3\text{I}$  and water molecules were adsorbed. Then the water molecules in pores acted to enhance the chemical reaction between  $\text{CH}_3\text{I}$  and the tertiary amine. According to DFT calculation [25], the heats generated by the alkylation reaction of  $\text{CH}_3\text{I}$  with QUID and TEDA were estimated 20.0 kJ/mol and 12.8 kJ/mol, respectively. Therefore, if  $\text{CH}_3\text{I}$  reacts with the impregnants in the pore, it cannot diffuse into extremely deep pores where the adsorption potential is high. In case of tertiary amine up to 5 wt% loaded on the surface and pore of AC, more  $\text{CH}_3\text{I}$  molecules are preferentially chemisorbed on the modified activated carbons. Experimental results showed that the QUID-impregnated AC had a much higher  $\text{CH}_3\text{I}$  removal efficiency than the TEDA-impregnated AC at the nearly same amount of impregnant. It seems that QUID can be used as a promising chemical agent for removing radioactive methyl iodide.

## 4. Conclusion

The trapping capacity of radioactive methyl iodide with two different tertiary amine loaded ACs were evaluated at a high humidity up to 95% by applying ASTM D 3803. Both TEDA-AC and QUID-AC as adsorbents showed a good performance in removing methyl iodide (>99.9%) at a high humid condition up to 95% RH. Among these two different tertiary amines, QUID showed a higher trapping capability of radioactive methyl iodide at a high moisture feed stream condition up to 95% RH. It was considered that the chemical reaction of QUID with methyl iodide ( $\text{CH}_3\text{I}$ ) might be significantly enhanced due to the higher nucleophilicity than that of TEDA in the presence of a high humid condition. As for QUID-AC, the decontamination factor (DF) for removing radioactive methyl



iodide under a high humid condition was more than 1,000, and it satisfied the requirements of the regulation index for the iodine filters in nuclear power plants (NPPs). QUID can be used as a new chemical impregnant for removing the radioactive iodine generated from nuclear power plants (NPPs).

### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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