Characteristics of Uranium Dioxide (UO₂) Kernel Produced by Sintering Process using Modified Sintering Reactor

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Abstract. Sintering process is the final stage of fuel kernel manufacturing prior to the coating process. This stage is very important part of the whole process, because it will determine the feasibility of UO₂ kernel to comply with the specifications of HTR reactor fuel. The objective of this research was to obtain UO₂ kernel with the density of ≥ 95% TD. The results showed that the highest density reached 92.56% TD or about 10.1441 g / cm³. This condition of sintering was gained at the temperature of 1400 °C with sintering time of 2 hours. The sintering product diameter gained was around 919 μm, the specific surface area 4.4213 m² / g, and a total pore volume 4.751 x10⁻³ cm³ / g. The density of UO₂ kernel produced from this research is the best compared to previous finding because of its density already approaches the HTR fuel specification requirements

Keywords: density, sintering, UO₂ kernel, reactor fuel, HTR

I. Introduction

The utilization of nuclear energy in Indonesia has become one of the highly prospective alternative energy besides fossil and renewable energy. Various studies have been done in an effort to prepare for the construction of Nuclear Power Plant (NPP) [1-2]. Generating prospective development is the type of high-temperature reactor. High-Temperature Reactor (HTR) is a privilege type of reactor having a reliably secure and highly thermal efficient. In terms of safety, HTR uses helium gas as the cooling material which is one phase and an inert fluid, fuel in the form of coated particles capable confining fission reactions at high temperatures, graphite with a high heat capacity as the moderator, as well as the reactor core made of ceramic material capable of surviving the high temperature rise. HTR also has a negative reactivity on overload the reactor would shut down automatically when the temperature rises excessively [3-5]. The use of graphite as a moderator and helium gas as the cooling material, is able to produce a higher heat so that it can be utilized a heat source for industries such as chemical process industry, the production of hydrogen, coal gasification, and desalination [6-8].

The status of HTR fuel research development at the moment has reached the stage of preparation of fuel elements which includes solution preparation sol, gelation process, aging, drying, calcination, reduction, sintering, coating and molding kernel. The series of studies aimed at getting HTR fuel in order to comply with the defined specifications [2,3,9]. Various studies related to HTR fuel preparation in Indonesia have been carried out until sintering phase. However, the kernel produced has not met the required specification. The produced kernel density remains below the required specification of less than 95% theoretical density (UO₂ Theoretical Density = 10.96 g / cm³).
Therefore, furthermore research needs to be conducted to get the kernel product which comply with the required quality of HTR fuel. Some researchers [10] conducted research previously on the effect of temperature and sintering time on the quality of UO$_2$ fuel kernel by using the fluidized bed furnace type in an argon gas atmosphere, the produced kernel density of approximately 91.136% of the theoretical density. Other researchers [11] conducted further research on sintering of UO$_2$ kernel by using fluidized bed furnace in the argon g medium as at a temperature of 1000 °C-1150 °C. The density of UO$_2$ kernel product obtained was 9.99 g / cm$^3$ (91.15% of the theoretical density). Those above kernel products did not comply with the HTR fuel standards. China has also conducted research on the preparation of UO$_2$ fuel kernel HTR-10. The sintering process was carried out in the research by using hydrogen gas atmosphere at temperatures above 1500 °C. The produced kernel density reaches 98% theoretical density [12].

A few researcher conducted research focused on increasing UO$_2$ kernel density by using the inert gas atmosphere without mathematical modeling analyzing of sintering process. Therefore, this research intends to formulate mathematical modeling of sintering process in order to obtain optimum condition. The objectives of this research are: to get UO$_2$ fuel kernel with a high density which is ≥ 95% TD, to know the optimum conditions of UO$_2$ fuel kernel sintering process, to know the temperature and time effect on diameter, specific surface area, and total pore volume of UO$_2$ fuel kernel. This research is expected to yield UO$_2$ fuel kernel complying with a good quality HTR fuel specification.

Density and quality of kernels are largely influenced by the sintering temperature. Theoretically, the higher sintering temperature means the higher sintering process rate would leads the increasing of atoms mobility followed by the decreasing of particles pores so that the density of sintering product would be highly increases [13]. Research conducted by the Division of Nuclear Science and Technology at the Oak Ridge National Laboratory (ORNL), The United States. The higher the sintering temperature, the higher the rate of sintering process (solid sintered/time). Results of research conducted by the Division of Nuclear Science and Technology at the ORNL, the United States, showed that the sintering UO$_2$ for 5 hours at a temperature of 1550 °C in an atmosphere of hydrogen and argon (4% H$_2$ and 96% Ar) produces a quite high density kernel at around 98% of the theoretical density (10.82 ± 0.16 g / cm$^3$) [14].

Sintering time affect the quality of the kernel. Sintering time too quickly can allow the imperfections at sintering process product so that the density obtained has a lower value than that of the sintering product processed in quite a long time at the same temperature [15]. Kernel size affects the specific surface area of the final products of sintering. The smaller the size of the kernel the better level of densification on the material will be. Atmospheric sintering may affect the densification and the formation of microstructure in materials. If there was gas which is easily absorbed, it would disrupt the process of densification. Heating rate of sintering is the rising of sintering temperature which changes every time. Heating rate should be regulated in order to avoid thermal shock which can cause a breakdown of the kernel. Heating done periodically by raising the temperature from low temperature to high temperature so that the particles in the kernel can adjust the temperature changes that occur in a sintering furnace. The influence of U$_3$O$_8$ powder, formed by the various oxidation temperatures, on the sintered density of CANada Deuterium Uranium (CANDU)-type UO$_2$ pellet was investigated. Increasing of the oxidation temperature of U$_3$O$_8$ powder causes decreasing of the sintered density of UO$_2$ pellet [16-18].

2. Materials and Methods

2.1. Materials
The raw material used in this research was UO$_2$ fuel kernel. Fuel kernel of UO$_2$ was generated from the previous process of dissolution, sol-gel, soaking, drying, calcination and reduction. Specifications UO$_2$ kernel before sintering were a diameter of 1.015 mm, density of 8.1307 g/cm$^3$, the specific surface area 5.512 m$^2$/g, total pore volume of 0.0067 cm$^3$/g and the ratio of O/U 2.03. Argon gas is used as a medium or atmospheric sintering. Specifications argon gas is Argon UHP> 99.9995%, O$_2$ <2 ppm, H$_2$O <3 ppm.
Density of UO₂ fuel kernel analysis use ABM (demineralized water) of PSTA (Center for Science and Accelerator Technology) production and CCl₄ that can be obtained from local market.

2.2. Experimental Procedure
Kernel UO₂ was put into the furnace (which has been previously vacuumed and then filled with argon gas as the medium) at a certain temperature (hereinafter the temperature was varied in accordance with the experimental design). The heating process of UO₂ kernel within the furnace was done in several stages of preparation. The process of heating covers heating rate in particular, certain fixed temperature (the sintering temperature was varied at 1300 °C, 1400 °C and 1500 °C), for a certain time (the sintering time was varied according to experimental design namely time variables were 1, 2, 2.5 and 3 hours). Meanwhile, in the cooling stage: lower heating temperature approaching the ambient temperature as well as the final termination argon gas flow and the flow of cooling water. Further analysis of kernel includes the products of the UO₂ kernel sintering process. Diameter optical microscope dinolite capture 2.0, UO₂ kernel density with a pycnometer and advance comprehensive analysis of the specific and total pore volume of kernel using NOVA-1000.

2.3. Experimental Apparatus
A series of equipments used in this research are shown in Figure 1

![Figure 1. Sintering process equipment scheme.](image1)

![Figure 2. Experimental apparatus.](image2)
3. Results and Discussion

3.1. Temperature Profile

The overall research investigating the effect of sintering temperature and time of changing the density, diameter shrinkage, changes in the specific surface area and total pore volume. Variables of the investigated temperature was 1300 °C, 1400 °C and 1500 °C. Variable of time was 1; 2; 2.5, and 3 hours. The ratio O/U of UO₂ kernel was considered fixed because it was used an inert argon gas atmosphere so that there was no oxidation. The kernel of UO₂ used was the reduction product of U₃O₈ at a temperature of 800 °C - 850 °C with a density of 8.1307 g/cm³. The rate of heating could heavily influence the sintering process, in addition to the temperature and time of sintering. Research temperature profile was shown in Figure 2 and Figure 3. Compare with the temperature profile of ORNL research [14], the heating rate of which was different. Heating rate of UO₂ kernel sintering product in this investigation was not constant. Average heating rate ranged from 8.04 to 9.02 °C/min. The temperature profile of other research conducted by Oak Ridge National Laboratory (ORNL) was shown in Figure 5.

In Figure 5, The ORNL researchers showed their experimental data of retention time at a certain temperature before reaching the maximum temperature while in this experiment showed no retention time data. This gives a different effect on the final sintering products. Retention time before the maximum temperature allow a sufficient time for the atomic particles to restructure properly, by which the pores would be reduced significantly so that the kernel density sintering products may approach the theoretical density. These results indicate that the density values obtained lower than the density of the results of sintering at ORNL. Although the temperature profile factor is not the only factor that causes the kernel density difference sintering results, but the temperature profile factor is an important factor in the success of the sintering process.

![Figure 3. Heating rate of 1300 °C, 1400 °C and 1500 °C.](image)

![Figure 4. Profile temperatures at various times on UO₂ kernel sintering](image)

![Figure 5. Profile temperatures at various times on UO₂ kernel sintering at ORNL [14]](image)
3.2. Effect of Temperature on Density

Data effect of temperature on UO$_2$ kernel density changes are shown in Table 1 and Figure 5.

| Temperature (°C) | Density UO$_2$ achieved (g/cm$^3$) | Relative Density (% TD) |
|------------------|------------------------------------|-------------------------|
| 0                | 8.1307                             | 74.19                   |
| 1200             | 8.7998                             | 80.29                   |
| 1300             | 9.3378                             | 85.20                   |
| 1400             | 10.1441                            | 92.56                   |
| 1500             | 10.0341                            | 91.55                   |

From Table 1 and Figure 6 show that the UO$_2$ kernel density increases along with increasing the sintering temperature that is at a temperature of 1300 °C to 1400 °C, and then it remains constant at a temperature of 1500 °C. From the measurement results show that kernel density at a temperature of 1500 °C is lower than that of kernel density at a temperature of 1400 °C. But the value of the density reduction is still relatively small at 0.11 g/cm$^3$, or about 1.084%. The increase in density caused by the densification during the occurring of sintering process. At high temperature, partial melting process occurs inside the particles. Open pores which is initially contain air started coming out and interconnected to form a closed pore so that the solid’s structure becomes more dense; the pore volume to be reduced in line with the increase in sintering temperature.

The high density of the kernel sintering products due to the densification predominate compared with coarsening processes. Conversely, if the coarsening process dominates the density of UO$_2$ kernel sintering products will decrease. It is due to an equilibrium pressure of gas in the pores (closed pore) so that the densification process stops. Gas trapped in the pores affect the pore elimination rate. Gas trapped in the closed pores undergoes expansion due to high temperature. This causes the kernel density to decrease.

This study resulted in a lower density than that of the previous study [12,14,19]. That is because the operating condition of sintering and samples of different raw materials. The temperature profile at different times and different heating rates have an impact on the achievement of different final densities. Sintering temperature profile in the study increases continuously and no detention time at certain temperature because there is no detention facility at the temperature of the equipment used.

Detentions can only be obtained when the temperature reaches the maximum temperature. Differences in temperature versus time profile can be seen in Figure 4 and Figure 5. The detention period at a certain temperature is intended to give an opportunity to the structure of particles in performing the
reduction or elimination of the pores. Samples of different raw materials also provide a different final outcome of sintering. Diameter of kernel in this study approximately 1.0 mm (10³μm) is greater than the diameter of the kernel on the experimental data of Collins et al which is 500 ± 20 μm [14]. Theoretically that the larger the particle size so that the lower relative density would be achieved. This is consistent with the results of this investigation namely with larger diameter than that of diameter investigated on the previous investigation [14], hence the density yielded in this investigation was lower which is only about 92% TD. Research on the sintering UO₂ kernel in the medium NH₃ was also conducted by [18]. The results showed the increase density of sintering kernel along with increasing sintering temperature and reaches a maximum at a temperature of 1400 °C with the results of the density of around 95% TD. Graph of the correlation between the density and temperature of sintering UO₂ kernel research results [19] shown in Figure 7.

3.3. Effect of Time on Density

Data the effect of time on the density of UO₂ kernel sintering products are shown in Table 2 and Figure 7.

| Time (hour) | UO₂ Density achieved (g/cm³) | Relative Density (%.TD) |
|------------|------------------------------|------------------------|
| 1          | 9.6830                       | 88.35                  |
| 2          | 10.1441                      | 92.56                  |
| 2.5        | 9.8066                       | 89.48                  |
| 3          | 9.4154                       | 85.91                  |
| 4          | 8.9400                       | 81.57                  |

Figure 8. Correlation of relative density of UO₂ kernel at various time.

From Table 2 and Figure 8 shows that the kernel density of UO₂ increases with increasing sintering time up to 2 hours, the increase of UO₂ kernel density up to 92.56% TD and sintering time after 2 hours decreases the density up to 81.57% TD. Based on the graph relation between relative density and sintering time (sintering stage theory), the time between 0 to 2 hours is intermediate sintering, where the pores in the particles are connected so that the solid structure becomes denser and the solid density becomes higher. Sintering time provides enough opportunity for atoms to restructure atoms. Conversely, if the sintering time is more than 2 hours, the pores become isolated and when the sintering time increases, the isolated pores become larger. This causes the kernel density to decrease.

In this investigation, early kernel density was 8.1307 g/cm³, the target density was ≥10.4 g / cm³ (95% TD), density reached was 10.1441 g/cm³ (92.56% TD). Achievement of UO₂ kernel density
sintering results in this study is good because the minimum limit value of the required density of 92.56% TD was gained. That is, it takes only about 2.46% to comply with any kernel density qualification with HTR fuel specifications. Some reasons that the achievement of a kernel density sintering products is still slightly below specification HTR due to the heating rate is so fast and is unstable. Additionally, in this investigation also there was not any detention time at a certain temperature. The temperature increased linearly and detention time was carried out at a maximum temperature. The ratio O/U targets are $1.99 \leq x \leq 2.02$, and the reached ratio O/U was 2.03. The ratio O/U related to the initial manufacturing process of UO$_2$ kernel from preparation urania sol solution, gelation, soaking, to heating up the kernel. Change in the ratio O/U could be caused by the occurring of oxidation processes during the preparation process prior to the sintering. Therefore, it would be much better if the reduction and sintering processes were integrated. The completed UO$_2$ kernel should be reduced in the reduction furnace followed by sintering stage at the same furnace. An atmospheric gas used during sintering should be the mixture of Ar and H$_2$ with a composition of 4% H$_2$ and 96% Ar in which the residual oxygen could be reduced in order to completely reduction process.

3.4. Effect of Temperature and Time on Change Diameter
Data effect of temperature and time on the change diameter of UO$_2$ kernel sintering products is shown in Table 3 and Figure 9.

| Time (hour) | UO$_2$ Kernel Diameter (mm) |
|------------|-----------------------------|
|            | 1300°C  | 1400°C  | 1500°C  |
| 0          | 1.015   | 1.015   | 1.015   |
| 1          | 1.013   | 1.003   | 0.940   |
| 2          | 0.988   | 0.919   | 0.920   |
| 2.5        | 0.986   | 0.931   | 0.954   |
| 3          | 0.991   | 0.940   | 0.945   |

Figure 9. Correlation of Relative Diameter of UO$_2$ kernel at various temperature and time.

From Table 3 and Figure 9, it was written that kernel diameter decreases very sharply at a time under 2 hours, reaching 0.919 cm at the heating temperature of 1400 °C. This happened almost at the same temperature of 1300 °C, 1400 °C and 1500 °C. After the kernel diameter has increased over 2 hours. The reduction of Kernel diameter is due to a reduction in volume and pore size while an increase in diameter indicating the existence of grain growth in the particles. In addition to the occurring of grain growth, the increase of the diameter size of the kernel could also be caused by the expansion of gas trapped in the closed pores as the result of increasing sintering temperature and time.

3.5. Effect of Temperature and Time on the Specific Surface Area of UO$_2$ kernel

Figure 10. Correlation of SSA of UO$_2$ kernel at various temperature and time.
Data influence of sintering temperature and time of the UO$_2$ kernel specific surface area is shown in Table 4 and Figure 10.

Table 4. Effect of temperature and time on specific surface area of UO$_2$ kernel.

| Time (hour) | Specific Surface Area (m$^2$/g) |
|------------|---------------------------------|
|            | 1300°C  | 1400°C  | 1500°C  |
| 0          | 4.9424  | 4.9424  | 4.9424  |
| 1          | 4.9445  | 4.6356  | 4.7389  |
| 2          | 4.8431  | 4.6761  | 4.4213  |
| 2.5        | 4.9475  | 4.6097  | 4.6798  |
| 3          | 4.9187  | 4.6893  | 4.6708  |

Table 4 and Figure 10 shows that the higher the sintering temperature, also the higher the decrease of specific surface area. Decline of specific surface area was due to the increasing of contact point between the crystal structure inside the particles and the reduction of pores distribution inside the particles. The decrease of specific surface area greatly contributes to the sintering process due to the change in the particles free energy that encourage the occurring of densification.

In the beginning of sintering at the time of less than 2 hours the surface area decline happened, but at the sintering time over 2 hours the specific surface area increases. The increase in specific surface area indicates that the kernel is getting more porous. This is also proven by the decrease value of kernel density after sintering time of 2 hours.

In this investigation the initial surface area was 5.512 m$^2$/g, target surface area was 3.11 m$^2$/g, and the reached surface area was 4.421 m$^2$/g. Total pore volume initially was 0.0067 cm$^3$/g, total pore volume target of 0.001 cm$^3$/g, and a pore volume achieved was 0.0044 cm$^3$/g. Achievement of surface area reduction and total pore volume in this investigation reached 0.45% and 0.40%. This achievement is quite good, but still needs to be improved. Surface area is closely related to the distribution and pore size inside the kernel.

Achievement of surface area and total pore volume which are still below the prerequisite specifications due to the existence of pores inside the kernel. These pores formation was commenced from the gel formation process. The existence of air trapped in the gelation process causes the formation of closed pores inside the kernel. The sintering process which is not optimum could also be the cause of the reduction in distribution and pore volume less than the maximum. Data the influence of temperature and sintering time on the total pore volume are presented in Table 5 and Figure 11.

Table 5. Effect of temperature and time on total pore volume of UO$_2$ kernel sintering results.

| Time (hour) | Total Pore Volume (cm$^3$/g) x 10$^3$ |
|------------|----------------------------------|
|            | 1300°C  | 1400°C  | 1500°C  |
| 0          | 6.7316  | 6.7316  | 6.7316  |
| 1          | 5.5627  | 5.1708  | 4.9203  |
| 2          | 5.3153  | 4.7515  | 4.8275  |
| 2.5        | 5.8522  | 5.4634  | 5.3422  |
| 3          | 6.0834  | 5.4829  | 4.8846  |
Initially, all the pores inside the particles were separated one another then when the sintering happened those pores would be interconnected. During sintering, the open pores turn into a closed pore. Effect of sintering time on the porosity of UO$_2$ sintering are shown in the graph in Figure 10. At a temperature of 1400 °C, total pore volume of UO$_2$ kernel decreases from $6.7316 \times 10^{-3}$ cm$^3$/g to $4.7515 \times 10^{-3}$ cm$^3$/g. then the total pore volume increases after sintering time up to 2 hours. research results in figure 10 show the similar phenomenon to that results of research conducted by [12] shown in figure 11. From Figure 11 and Figure 12 it is shown that the total porosity of UO$_2$ decreases at initial time of sintering caused by the decrease of the open pore volume which is turns into a closed pore, while the closed pore increases continuously and decreases again after reaching its peak.

The phenomenon of open pore decrease in the beginning of sintering occurs due to the release of gases in the pores and the occurring of material diffusion and atomic restructuring led to the more densely structure, while, the continuously improvement in closed pores caused by the expansion of gas trapped in the pores due to the increase in the sintering temperature so that the pore size also increases. the pore size does not increase and tends to fall when it reaches the optimum time and temperature because of the occurring of the equilibrium between the gas pressure inside the pores with the pressure outside the pore. The ongoing material diffusion would gradually reduce the pore distribution so that the distribution and pore size decrease.

4. Conclusions
From the research and discussion above, it could be concluded that the highest density of UO$_2$ kernel sintering products obtained was 10.1441 g/cm$^3$ (92.56% TD). Achievement of UO$_2$ kernel density of sintering products was good because it was close to the minimum required density values of HTR fuel specification (HTR fuel kernel density is ≥ 95% TD). The optimum condition was achieved at a temperature of 1400 °C with an isothermal sintering time for 2 hours. The resulting kernel diameter was about 919 μm, the specific surface area was 4.421 m$^2$/g, and a total pore volume 4.751 x 10$^{-3}$ cm$^3$/g.

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