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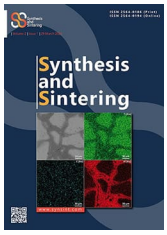

*Research article*

# Molecular hydrogen production by radiolysis of water on the surface of nano-$ZrO_2$ under the influence of gamma rays

**Gunel Imanova**

*Institute of Radiation Problems, Azerbaijan National Academy of Sciences, AZ 1143-Baku, Azerbaijan*

ABSTRACT

In this research, the radiation-heterogeneous processes of water decomposition on the surface of zirconium dioxide nanoparticles (n-$ZrO_2$) were studied. The kinetics of buildup of molecular hydrogen during the radiolytic processes of water decomposition was also examined. The production of $H_2$ and $H_2O_2$ through water radiolysis was investigated to develop a computational model and disclose the kinetic behavior of water radiolysis. The enthalpy of $ZrO_2$ nanoparticles was studied at the temperature range T=1200–2900 K, in which $ZrO_2$ nanoparticles has a two-phase transition. Some of the electrons were transported to the surface of the nanoparticles during the physical and physicochemical stages of the process and emitted into the water. At the same time, the migration of energy carriers in radioactively active oxide compounds changed at different intervals depending on the composition, structural stability, and electro-physical properties of the oxides.



KEYWORDS

Hydrogen generation
Nano zirconium oxide
Enthalpy
Water splitting
γ-radiation

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## 1. Introduction

The radiolysis of water results in the production of electrons, atoms, radicals, ions and molecules due to ionizing radiation. A brief history of the development of the understanding of water radiolysis with a focus on the $H_2$ production has been published [1–2]. It was found that the energy carriers' migration in radiation-catalytic active oxides varies at different intervals depending on the oxides composition, their structural regularity and electrophysical characteristics. Therefore, the effective energy transfer distance in radiation-catalytic active oxides is limited by these interval dimensions, which in turn shows the radiation-catalytic activity, which again depends on the particle size [3–8].

Nano-$ZrO_2$ has good thermal insulation and dielectric properties over a wide temperature range, which allows it to be considered as a promising material in the field of microelectronics and the use of construction materials [9–14]. Nanostructure materials possess developed surfaces and increased defects on the boundary of particles, which are of remarkable significance in radiation-heterogeneous processes with their participation, and during the progression of highly sensitive detectors of ionizing radiation [15–18]. Heating devices operate at fairly high temperatures, using nanoparticle $ZrO_2$ to ensure their high quality. However, when using nanoparticle $ZrO_2$ as a construction material, it is necessary to take into account the large number of phase transitions at high temperatures.

In this work, the amount of molecular hydrogen obtained from radiolysis processes by changing the amount of water (m=0.02, 0.04, 0.06, 0.08, and 0.10 g), its formation rate, and radiation-chemical emissions are studied in the nano-$ZrO_2$/$H_2O$ systems with d=20–30 nm particle size under the impact of gamma rays ($^{60}Co$, $D_\gamma$=0.30 Gy/s, T=300 K).

## 2. Experimental

The nanosized zirconium dioxide with 99.9% purity, diameter of 20–30 nm, and density of 0.4–0.6 g/cm$^3$ was purchased from SkySpring Nanomaterials, Inc., USA. The analysis of hydrogen was carried out using the Agilent-7890A chromatograph in the following mode: column: C-1010 P., 30 m×0.53 mm I.D. (25467), D.-TCD,

* Corresponding author. *E-mail address:* radiasiya555@rambler.ru (G. Imanova)

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F.-3 ml/min, 20 Hz/0.01 min, T(head)=50 °C, T(d.)=230 °C, M.F.=0 ml/min, g. C.–Ar, oven: 50 °C (7.0 min), 20 °C/min to 230 °C, i.T.: 230 °C.

The radiation test was done employing the gamma $^{60}Co$ isotope source. The retained measurements rate of the source was dDγ/dt=0.40 Gy/s. The calculation of the radiation dosage in these frameworks was performed in comparison with the electron thickness [19–21].

## 3. Results and discussion

Initially, the enthalpy of nanoparticle $ZrO_2$ (M=123.22 g/mol) was considered in the temperature range T=1200–2900 K. It is known that nanoparticle $ZrO_2$ has a two-phase transition in the temperature range under study. In the phase transition region, samples should be kept under the study temperature for about 1 hour to ensure the phase transition of the entire mass of the substance. When measuring the enthalpy of nanoparticle $ZrO_2$, it should be borne in mind that the samples undergo two structural transformations. When the samples are heated in the furnace above the transition temperature, the process of complete and partial conversion from one structure to another takes place, and when the samples fall into the calorimeter, a reverse transition is observed, resulting in heat dissipation during cooling. Thus, according to this method, the phase transitions are fixed during the cooling of the samples. The obtained results are shown in Fig. 1, where the average heat capacity varies with temperature. Experience has shown that the enthalpy creates three conditional temperature zones that differ in velocity: (I: 1200–1450 K, II: 1450–2650 K, III: 2650–2900 K).

From this, we can conclude that the $ZrO_2$ nanoparticles have the structure of momoclinic in the first region, as a result of which it undergoes a complete tetragonal modification in the temperature range of region I. The change in the growth rate of enthalpy in the temperature range of region II is due to the polymorphic transformation of the tetragonal structure of $ZrO_2$ nanoparticles into a cubic structure. In this regard, when processing the obtained results, it should be noted that in the temperature range $T_I \le T_{II} \le T_{III}$ the $ZrO_2$ nanoparticles consist of a two-phase (monoclinic + tetragonal) state, which also depend on the temperature.

The experimental values of zirconium dioxide nanoparticles for enthalpy over the entire temperature range are given by a single equation that takes into account the structure of the sample:

$$H_T\text{-}H_{298.15}=(1\text{-}K_1)\,H^m(T) + K_1(1\text{-}K_2)\,H^t(T) + K_2H^c(T) \quad (1)$$

where $H^m(T)$, $H^t(T)$ and $H^c(T)$ are lines of enthalpy regressions for monoclinic, tetragonal and cubic modifications, respectively. The coefficients $K_1$ and $K_2$ (concentrations of tetragonal and cubic structures) have the following values:

$K_1$=0 for monoclinic modification;

$K_1$=1 for tetragonal and cubic modification;

$K_1$=[($T_1$-$T_1$-)/($T_1$+-$T_1$-)] for solutions of monoclinic and tetragonal modifications;

$K_2$=1 for cubic modification;

$K_2$=0 for other modifications.

The heat capacity of monoclinic, tetragonal, and cubic modifications is given by the following equations, respectively:

$$C_P^m = a_0 + a_1T + a_2T^2 + a_3\left[1/T^2\right] \quad (2)$$

$$C_p^T = a_4 + a_1T + a_5T + a_6T^2 \quad (3)$$

$$C_p^K = a_7 + a_8T + a_9T^2 \quad (4)$$

and taking into account (1), we obtain the following equation for the average heat capacity in the entire temperature range studied (5):

$$C_p = (1-K_1)\left[a_0 + a_1(T+T_0)/2 + ((T^2+T_0T+T_0^2)/3 - a_3/T_0T)\right] +$$
$$K_1(1-K_2)\left[a_4 + a_5(T+T_0)/2 + a_6(T_2+TT_0+T_0^2)/3\right] +$$
$$(1-K_2)\left[a_4 + a_5(T+T_0)/2 + a_6(T^2+TT_0+T_0^2)/3\right] \quad (5)$$

The coefficients of Eq. 5 were calculated by the least squares method. The obtained values show that at $T_1$+-$T_1$-=9 K the transition from one structure to another takes place at the temperature $T_1$=1450 K.

According to the calculations, the conversion from tetragonal modification to cubic modification takes place at $T_2$=2660K and the constants of Eq. 5 are presented below (cal/mol for enthalpy): $a_1$=6.94045×10, $a_2$=8.79621×$10^{-3}$, $a_3$=-1.43165×$10^6$, $a_4$=7.52967×10, $a_5$=3.55216×$10^{-3}$, $a_6$=4.93324×10, $a_7$=2.39006×$10^{-2}$ and variance of measurement results $S_0^2$ =0.589.

The results of research in the temperature range T=1200–2800 K allowed us to find the value of the heat of polymorphic transformations: -5.5±1.5 kcal/mol and -8.6±1.5 kcal/mol, respectively 6.5% and 4.5% enthalpy size occurs at the transition point. The enthalpy of conversions from monoclinic to tetragonal, from tetragonal to cubic modification is 3.8 and 3.2 kcal/mol, respectively.

Fig. 2 shows the irradiation time (dose) dependencies of the content of molecular hydrogen achieved from radiation-heterogenous decomposition of nano-zirconia (with varying mass, m=0.02, 0.04, 0.06, 0.08, and 0.10 g) in the nano-$ZrO_2$ system (paticle size d=20–30 nm) irradiated with gamma rays ($^{60}Co$, $D_\gamma$=0.30 Gy/s, T=300 K).

As shown in Fig. 2, the speed of the process remains almost constant as the amount of nano-$ZrO_2$ increases. This again shows that the processes occur on the surface regardless of the amount of catalyst. In this way, the surfaces of oxides are charged emphatically and have an electron-acceptor characteristic [22–26].

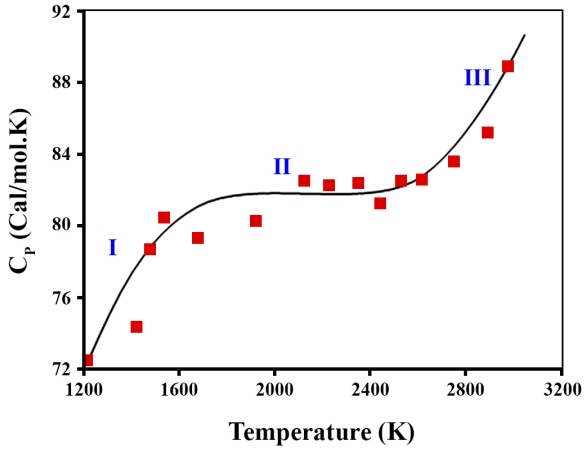


**Fig. 1** Average heat capacity of zirconium dioxide versus temperature.

The formation rate of molecular hydrogen was determined by water, nano-$ZrO_2$, and overall system from linear parts of kinetic curves (Fig. 2) of studied (nano-$ZrO_2$/$H_2O$) systems. Molecular hydrogen formation rate obtained by radiolysis of nano-$ZrO_2$ was determined based on the Eq. 6:

$$w_0(H_2) = 0.01G_0(H_2)P = \frac{N_0(H_2)}{m_{ZrO_2}t} \quad (6)$$

Fig. 2 shows the formation rate of molecular hydrogen from the kinetic part of the curves according to the nano-$ZrO_2$ in the nano-$ZrO_2$/$H_2O$ system.

$$w_{ZrO_2}(H_2) = \frac{N(H_2)}{m_{ZrO_2}t} \quad (7)$$

According to overall $ZrO_2$/$H_2O$ system:

$$w_{tot}(H_2) = \frac{N(H_2)}{m_{tot}t} = \frac{N(H_2)}{(m_{H_2O} + m_{ZrO_2})t}$$

$$= \frac{m_{ZrO_2}}{m_{H_2O} + m_{ZrO_2}} \frac{N(H_2)}{m_{H_2O}t} = \frac{m_{ZrO_2}}{m_{H_2O} + m_{ZrO_2}} m_{ZrO_2}(H_2) \quad (8)$$

According to nano-$ZrO_2$, considering the $\Delta N(H_2) = N(H_2) - N_0(H_2)$ increase in molecular hydrogen with the addition of nano-$ZrO_2$ was determined using the following equation:

$$w_{ZrO_2}(H_2) = \frac{\Delta N(H_2)}{m_{ZrO_2}t} = \frac{N(H_2) - N_0(H_2)}{m_{ZrO_2}t} \quad (9)$$

If we multiply the numerator and denominator of Eq. 9 by $m_{ZrO_2}$, with simple transformations considering the Eq. 6 and 7:

$$w_{ZrO_2}(H_2) = \frac{m_{ZrO_2}}{m_{H_2O}} \frac{N(H_2) - N_0(H_2)}{m_{ZrO_2}t} = \frac{m_{ZrO_2}}{m_{H_2O}} \frac{N(H_2) - N_0(H_2)}{m_{ZrO_2}t} =$$

$$= \frac{m_{H_2O}}{m_{ZrO_2}} \left( \frac{N(H_2)}{m_{ZrO_2}t} - \frac{N_0(H_2)}{m_{ZrO_2}t} \right) = \frac{m_{H_2O}}{m_{ZrO_2}} \left[ w_{ZrO_2}(H_2) - w_0(H_2) \right] \quad (10)$$

where, $G_0(H_2)$=0.45 molecule/(100 eV) is radiation-chemical yield of molecular hydrogen obtained from radiolysis of pure water and $N_0(H_2)$ and $N(H_2)$ is the content of molecular hydrogen obtained from pure water radiolysis and nano-$ZrO_2$/$H_2O$ system, respectively.

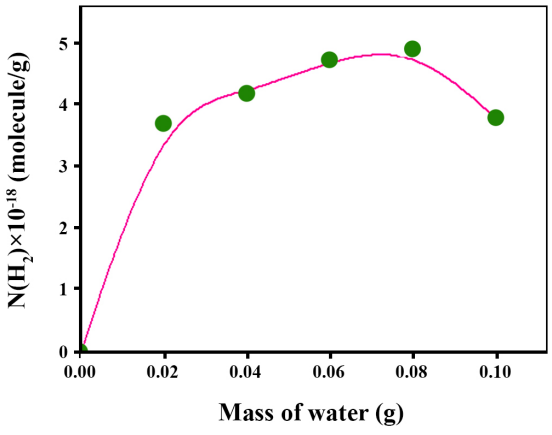


**Fig. 2.** Irradiation time (dose) dependencies of the content of molecular hydrogen achieved from radiation-heterogeneous decomposition of nano-zirconia (with varying mass, m=0.02, 0.04, 0.06, 0.08, and 0.10 g) in the nano-$ZrO_2$ system (paticle size d=20–30 nm) irradiated with gamma rays ($^{60}Co$, $D_\gamma$=0.30 Gy/s, T=300 K).

Therefore, Compton scattering mainly occurs compared to other processes (photoeffect, photonuclear reaction, electron-positron pair formation, etc.) in the $ZrO_2$, $H_2O$, $ZrO_2$/$H_2O$ systems under the influence of $E_\gamma$=1.25 MeV ($^{60}Co$) energy of gamma rays. The energy of Compton electrons varies in the range of 0–1.02 eV based on the scattering angle. The Compton electrons, on the basis of their kinetic energy, pass from the nanoparticle into the liquid phase or vice versa several times, losing the kinetic energies gradually in both elastic and inelastic collisions and turning into thermal electrons in nano-zirconia/$H_2O$ systems. In an inelastic collision in the physical phase ($10^{-15}$–$10^{-12}$ s) of the process: 1) electron ($e^-$)-hole ($ZrO_2^+(h^+)$) pair and excited electron of $ZrO_2^*$ from molecules direct single ionization within nano-zirconia:

$$ZrO_2 \xrightarrow{\gamma} ZrO_2^+(h^+), ZrO_2^*, e^- \quad (11)$$

2) electron-ion ($H_2O^+$) pair and excited electron of $H_2O^*$ from molecules direct single ionization in water:

$$H_2O \xrightarrow{\gamma} H_2O^+, H_2O^*, e^- \quad (12)$$

Active intermediate, unbalanced energy carriers are formed during Eqs. 11 and 12.

If the energy required to form an electron-hole pair inside $ZrO_2$ is under the impact of ionizing radiation (gamma rays, electrons) [21], then the radiation-chemical yield of the electron-hole pair is $G(h^+\text{-}e^-)$, the radiation-chemical yield of the electron-excitation (exciton) states is equal to $G_{exc}$. These active intermediates ($h^+, H_2O^+, e^-, ZrO_2^*, H_2O^*$) play an important role in the process of achieving molecular hydrogen from the radiation-heterogeneous decomposition of water in the zirconia/$H_2O$ system.

The holes generated inside the nanoparticle by ionizing radiation can be trapped by drift [22], part of which can be trapped by structural defects inside the particle, and another part can be transported to the particle surface and trapped by an adsorbed complex of water [$ZrO_2$–$H_2O_s$] on the surface and formed ion-complex as follow:

$$\left[ZrO_2 - H_2O_s\right] + h^+ \rightarrow \left[ZrO_2 - H_2O_s\right]^+ \quad (13)$$

This ion-complex causes to electron-excitation of the complex by recombination with heat or tunneling electrons:

$$\left[ZrO_2 - H_2O_s\right]^+ + e^- \rightarrow \left[ZrO_2 - H_2O\right]^* \quad (14)$$

On the other hand, the excitons formed by ionizing radiation can be absorbed inside the nanoparticle and transmit their energy to a water complex adsorbed on the surface at a distance. In this case, the electronic-excitation of the complex is observed:

$$exc + \left[ZrO_2 - H_2O_s\right] \rightarrow \left[ZrO_2 - H_2O\right]^* \quad (15)$$

The energy of the short-lifetime electron-excitation complex [$ZrO_2$–$H_2O_s$]* [23] is transferred to the adsorbed water molecule and causes its decomposition, resulting in the formation of intermediate products $H^+$ and $OH^-$:

$$\left[ZrO_2 - H_2O\right]^* \rightarrow ZrO_2 + H^+ + OH^- \quad (16)$$

In order to obtain $H^+$ and $OH^-$ intermediate products from the decomposition of the water molecules, it is necessary to break the bond between them ($E_{rab}$). Therefore, the connection between the transmitted excitation energy ($E_{exc}$) and the communication energy must satisfy the $E_{exc} \geq E_{rab}$ condition.

The electrons radiated from the surface of the strong to fluid stage slowly lose their active vitality by versatile and inelastic collisions in water and initially changed over into warm electrons, and after that can rescue within the water [24–31]:

$$e^- + nH_2O \rightarrow e^-_{aq} \quad (17)$$

It has been proved both experimentally [25] and theoretically [26–31] in systems produced by suspension of nano-$ZrO_2$ in water that the radiation-chemical yield of salvaged electrons (Eq. 17) in the liquid phase is higher than in pure water, and this value varies depending on the size of the nanoparticles. The molecular hydrogen production during Eq. 18 and 19 by the radiolytic decomposition between water molecules and salvaged electrons ($e^-_{eq}$), as well as protonated water molecules ($H_3O^+$) in the intergranular liquid phase, can be described as follows:

$$2e^-_{aq} + 2H_2O \rightarrow H_2 + 2OH^- \quad (18)$$

$$2e^-_{aq} + H^+ + H_2O \rightarrow H_2 + OH^- \quad (19)$$

$$e^-_{aq} + H_3O^+ \rightarrow H^+ + H_2O \quad (20)$$

$$H + H \rightarrow H_2 \quad (21)$$

Here it becomes clear that one electron-hole pair or two excitons are used to obtain one molecule of hydrogen. Reactions 13–16 and 21 play a major role in the molecular hydrogen production from the radiation-heterogeneous decomposition of water adsorbed on the nano-zirconia surface under the influence of gamma rays. However, the radiation-chemical yield of molecular hydrogen obtained from the decomposition of water was less than 0.38 molecules/(100 eV) in systems created by the adsorption of water on the surface of nano-$ZrO_2$ irradiated by gamma rays. This implies that the surface thickness of the vitality exchange centers on the surface of nano-$ZrO_2$ is exceptionally little. As a result, the radiation-chemical surrender of atomic hydrogen produced during reactions 18–21 increases too much.

## 4. Conclusions

The results of research in the temperature range T=1200–2800 K allowed to find the value of the heat of polymorphic transformations: -5.5±1.5 kcal/mol and -8.6±1.5 kcal/mol, respectively 6.5% and 4.5% enthalpy occurs at the transition point. The enthalpy of conversions from monoclinic to tetragonal, from tetragonal to cubic modification is 3.8 and 3.2 kcal/mol, respectively. It can be concluded that radiation-chemical yield of molecular hydrogen obtained by radiolysis processes with varying mass of the nano-$ZrO_2$ in the range of $m_{ZrO_2}$ =m=0.02, 0.04, 0.06, 0.08, and 0.10 g in nano-$ZrO_2$/$H_2O$ systems (particle size-d=20–30 nm) irradiated with gamma rays ($^{60}Co$, $D_\gamma$=0.30 Gy/s, T=300 K) are as follow: if determined for nano-$ZrO_2$, the increase in $G(H_2)$=0.38–2.98 molecules/(100eV). Thus, in this way on the base of nanomaterials radiation-catalytic processes enable to transform ionizing radiation energy to chemical one with great efficiency. Nano size radiation-heterogeneous systems open new actual insight into radiation processes and radioactive study of materials. It is clear from the presented results that nanostructured materials become an important role in nuclear-power engineering as structural and functional materials practically in all stages of the nuclear fuel cycle. It is extremely important for the formation in nanostructured materials after exposure ordered nanostructure from the new phase with a period of a few nanometers, promoting the preservation of the properties of materials under high irradiation.

## CRediT authorship contribution statement

**Gunel Imanova:** Conceptualization, Formal Analysis, Investigation, Methodology, Supervision, Writing – original draft, Writing – review & editing.

## Data availability

The data underlying this article will be shared on reasonable request to the corresponding author.

## Declaration of competing interest

Although the author of this article is a member of the editorial board of *Synthesis and Sintering*, her role in the journal did not affect the reviewing process of the article.

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