Investigations on the Electrode Process of Concentrated V(IV)/V(V) Species in a Vanadium Redox Flow Battery

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Abstract: The electrode process of concentrated V(IV)/V(V) species has been studied at a graphite electrode by using cyclic voltammetry, low speed linear polarization, and impedance techniques. The results have revealed that in H₂SO₄ solution below 2 mol·L⁻¹ containing 2.0 mol·L⁻¹ V(IV), the electrode process of V(IV)/V(V) is controlled by the electrochemical polarization and diffusion with poor reversibility. When the concentration of H₂SO₄ is above 2 mol·L⁻¹, the electrode process of V(IV)/V(V) turns to diffusion control with improvement of the reversibility. An increase in concentration of H₂SO₄ solution facilitates a decrease in impedance, whereas too high concentration of H₂SO₄ solution (>3 mol·L⁻¹) results in a remarkable increase in the viscosity of solution leading to a large mass transportation polarization, and thus the impedance starts to increase a little. In 3 mol·L⁻¹ H₂SO₄ solution, the reversibility and kinetics of V(IV)/V(V) are improved gradually and the characteristics of impedance also improves with an increase in concentration of V(IV) solution. But, when the concentration of V(IV) solution exceeds 2.0 mol·L⁻¹, the viscosity of the solution is so high that the mass transportation polarization increases considerably, resulting in the deterioration of electrochemical performance of V(IV)/V(V) species and an increase in impedance. Therefore, by considering from the standpoint of increasing energy density and electrode kinetics comprehensively, the optimal concentration of H₂SO₄ is 3 mol·L⁻¹ containing 1.5–2.0 mol·L⁻¹ of V(IV).

Key Words: Vanadium redox flow battery; V(IV)/V(V); High concentration; Electrode process

Ever since Thaller¹ proposed the concept of the redox flow battery (RFB), researchers have been exploring possible redox couples. However, only recently, the sodium polysulfide/bromine and the vanadium redox battery have shown prospects of applications²-³. Effect of the cross-contamination of the positive electrolyte and negative electrolyte on performance of the RFB has been considerably diminished because of the use of a single element for both the negative and positive halves of the vanadium RFB. Therefore, the vanadium RFB is more superior to other systems.

Although performance of the vanadium RFB is influenced by many factors such as membranes and electrodes, investigations on the positive/negative electrode reaction kinetics are of the same importance for optimization of performance of the battery. So far, the V(IV)/V(V) electrode process kinetics have been described in a lot of literature, but concentration of the aqueous media used was limited to diluted vanadium solutions ranging from 0.1 to 0.5 mol·L⁻¹, which were far lower than that of practical electrolytes⁴-⁵. Based on spectroscopic and/or dynamic electrochemical data, the dissolved states of V(V) and V(IV) in concentrated sulfuric acid (H₂SO₄) solution were discussed in literatures⁶-⁷, whereas the conclusions kept rather different from one report to another. Oriji et al.⁸ studied the electrochemical behaviors of 2.0 mol·L⁻¹ VOSO₄ in 1–9 mol·L⁻¹ H₂SO₄ solution by applying a rotating disk electrode. Oriji et al.⁹ determined the electrochemical parameters such as diffusion coefficients and Stokes radii of V(V) and V(IV) species with high concentrations, finding that the electrode reaction of V(IV)/V(V) may be accompanied by a certain chemical step. But then, Oriji et al.¹⁰-¹¹ had employed
2 Results and discussion

In the present study, the electrode process kinetics of concentrated V(IV)V(V) at a graphite electrode have been studied by cyclic voltammetry, linear sweep voltammetry at a low scan rate, and impedance measurements to obtain the optimal concentration of V(IV)-H$_2$SO$_4$ solution used in the vanadium RFB.

1 Experimental

1.1 Preparation of V(IV)-H$_2$SO$_4$ solution

VOSO$_4$·nH$_2$O (Shanghai Lvyan Chemicals, n=3.0 according to thermogravimetric analysis, the purity is ≥97.9%) was dissolved in 1–4 mol·L$^{-1}$ H$_2$SO$_4$ to give a V(IV) solution.

1.2 Pretreatment of electrodes

A graphite rod (Shanghai Xinxia carbon Co Ltd) was employed as a working electrode. Prior to determination, the electrode surface was polished with a 1000-grit polishing paper, and then the electrode was sonicated for 10 min in water, followed by air-dry.

1.3 Apparatus and equipment

A Ubbelohde-type capillary viscometer; A model 600B electrochemical workstation (Shanghai CH Instruments Co.); An EG&G electrochemical workstation (Princeton Corporation, USA) including a 273A potentiostat and a 1025 frequency response analyzer.

1.4 Measurement procedures

The viscosity of the solution was obtained as follows: a volume of liquid was taken to flow through the capillary viscometer from a fixed height of the liquid surface in a thermostat bath of (20±2)°C, and the flowing time was recorded, multiplying with the correcting constant coefficient (0.04112 mm$^2$s$^{-1}$) to give the viscosity.

A graphite rod (area=0.103 cm$^2$) and a saturated calomel reference electrode(SCE) were employed as the working and the reference electrodes, respectively. Subsequently, a three-system was built by using a graphite plate with a large surface area as the counter electrode. The cyclic voltammetry and linear polarization plots of V(IV)-H$_2$SO$_4$ solution with different concentrations were determined by applying the model 600B electrochemical workstation. In the same cell with three electrodes, the impedance measurements of V(IV)-H$_2$SO$_4$ solution with different concentrations were carried out with an EG&G electrochemical workstation at frequency from 1×10$^5$ Hz to 0.1 Hz with an amplitude of ±5 mV.

2 Results and discussion

2.1 Effect of H$_2$SO$_4$ concentration on the V(IV)/V(V) electrode reaction

The concentration of V(IV) solution commonly used in the vanadium flow battery is 2.0 mol·L$^{-1}$.[5-6] Fig.1 shows cyclic voltammograms of 2.0 mol·L$^{-1}$ VOSO$_4$ in 1–4 mol·L$^{-1}$ H$_2$SO$_4$ solution. The potential differences($\Delta V_p$) and the ratios ($I_{pa}/I_{pc}$) of the anodic peak current to the cathodic peak current obtained from Fig.1 are listed in Table 1. As can be seen from Table 1, when applying 1 mol·L$^{-1}$ H$_2$SO$_4$, the anodic peak current is far higher than the cathodic peak current, leading to the $I_{pa}/I_{pc}$ beyond 1.8. It can be deduced from the electrode reaction expression (1) that 1.0 mol V(V) can not be reduced unless 2.0 mol H$^+$ exists in the solution. However, when 1 mol·L$^{-1}$ H$_2$SO$_4$ is used, the n(H$^+$) is just the theoretic value of H$^+$ needed for the reduction reaction of V(V). It suggests that practical requirements for the reduction reaction of V(V) would not be met. And hence the anodic peak current is much larger than the cathodic peak with a relatively larger potential difference and providing poor reversibility for the couple.

$$\text{VO}_2^++2\text{H}^++2\text{e}^-\rightarrow\text{VO}^2++\text{H}_2\text{O}$$ (1)

As the concentration of H$_2$SO$_4$ increases, the peak potential difference as well as $I_{pa}/I_{pc}$ decreases and the $I_{pa}/I_{pc}$ is gradually close to 1 with an improvement in reversibility of the

Fig.1 Cyclic voltammograms of a stationary graphite electrode in 1–4 mol·L$^{-1}$ H$_2$SO$_4$ solution containing 2.0 mol·L$^{-1}$ V(IV) scan rate=50 mV·s$^{-1}$, c(VOSO$_4$)(mol·L$^{-1}$): 1) 1; 2) 2; 3) 3; 4) 4

<table>
<thead>
<tr>
<th>c(H$_2$SO$_4$)(mol·L$^{-1}$)</th>
<th>$\Delta V_p$[a]/V</th>
<th>$I_{pa}/I_{pc}$[b]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.386</td>
<td>1.817</td>
</tr>
<tr>
<td>2</td>
<td>0.249</td>
<td>1.224</td>
</tr>
<tr>
<td>3</td>
<td>0.185</td>
<td>1.121</td>
</tr>
<tr>
<td>4</td>
<td>0.212</td>
<td>1.032</td>
</tr>
</tbody>
</table>

[a] $\Delta V_p$ means the difference between the anodic potential and the cathodic potential.
[b] $I_{pa}/I_{pc}$ means the ratio of the anodic peak current to the cathodic peak current.

Table 1 Cyclic voltamgram data of 2.0 mol·L$^{-1}$ VOSO$_4$ on a graphite electrode in 1–4 mol·L$^{-1}$ H$_2$SO$_4$ solution
couple. When the concentration of H$_2$SO$_4$ is up to 3 mol·L$^{-1}$, the potential difference is the lowest with the $I_0$/pc close to 1 and the peak current starts to decrease when using H$_2$SO$_4$ higher than 4 mol·L$^{-1}$. This is due to the fact that when the concentrations of H$_2$SO$_4$ are 1, 2, 3, and 4 mol·L$^{-1}$, the corresponding viscosities of the V(IV)-H$_2$SO$_4$ solutions are 7.895, 10.07, 10.65, and 12.62 mm$^2$·s$^{-1}$, indicating that when the concentration of H$_2$SO$_4$ is too high (>3 mol·L$^{-1}$), a relatively remarkable increase in viscosity can be observed and effect of mass transportation resistance on the electrode reaction becomes larger, resulting in a decrease in the peak current. Therefore, in the case of 2.0 mol·L$^{-1}$ V(IV) solution applied, it is favorable to employ 3 mol·L$^{-1}$ H$_2$SO$_4$ as a supporting electrolyte.

To explain the relationship between the concentration of H$_2$SO$_4$ and the V(IV)/V(V) electrode process, an overpotential of less than 25 mV is applied on the equilibrium potential of V(IV)/V(V). Also, a polarization resistance is determined by applying the sweep voltammetry at a low scan rate of 0.5 mV·s$^{-1}$. Fig.2 presents the typical linear polarization plot of 2.0 mol·L$^{-1}$ VOSO$_4$·1 mol·L$^{-1}$ H$_2$SO$_4$ solution.

In the potential range near the equilibrium electrode potential (≤25 mV), the relationship between current and overpotential is linear. The ratio of overpotential to current is called the polarization resistance ($R_p$) and related kinetic parameters calculated from the linear polarization plots of 2.0 mol·L$^{-1}$ VOSO$_4$ in 1–4 mol·L$^{-1}$ H$_2$SO$_4$ are listed in Table 2. The exchange current density ($I_0$) and reaction rate constant ($k_0$) were calculated using the following expressions$^{[12]}$, where $c$ is the concentration of the V(IV)-solution:

\[
I_0 = \frac{RTnF}{R_p} \quad (2) \\
I_0 = \frac{I_0}{nFc} \quad (3)
\]

As can be seen from Table 2, when the concentration of H$_2$SO$_4$ is 3 mol·L$^{-1}$, the polarization resistance of V(IV)/V(V) is the smallest with a high exchange current density and rate constant, which is consistent with the results of above cyclic voltammogram experiments. But, the rate constant data of V(IV)/V(V) obtained are two orders lower than those reported by previous workers$^{[6]}$. This difference may be due to that fact that the experiments reported here were carried out at extremely low overpotential limits$^{[12]}$.

### 2.2 V(IV)/V(V) electrode reaction- V(IV) concentration relationships

Using 3 mol·L$^{-1}$ H$_2$SO$_4$ as a supporting electrolyte, cyclic voltammograms of a stationary graphite electrode in 3 mol·L$^{-1}$ H$_2$SO$_4$ solution containing 0.3–3.0 mol·L$^{-1}$ V(IV) is presented in Fig.3. It can be seen that the cathodic peak current of 0.3 mol·L$^{-1}$ VO$\text{SO}_4$ solution is lower than the anodic peak current.

![Fig.2 Typical linear polarization plot of 2.0 mol·L$^{-1}$ VOSO$_4$·1 mol·L$^{-1}$ H$_2$SO$_4$ solution](image)

![Fig.3 Cyclic voltammograms of a stationary graphite electrode in 3 mol·L$^{-1}$ H$_2$SO$_4$ solution containing 0.3–3.0 mol·L$^{-1}$ V(IV) scan rate=50 mV·s$^{-1}$](image)
With an increase in concentration of VOSO$_4$, the cathodic peak current tends to be close to the anodic peak current, resulting in an improvement of reversibility. The peak current is enhanced sharply with the concentration of VOSO$_4$ increasing. When the concentration of VOSO$_4$ is 1.5–2.0 mol·L$^{-1}$, the peak current reaches the highest, whereas a further increase in the concentration of VOSO$_4$ leads to a decrease in the peak current, especially when the concentration of VOSO$_4$ is up to 3.0 mol·L$^{-1}$. It is seen from Fig.4 that the viscosity of the VOSO$_4$ solution above 2.0 mol·L$^{-1}$ increases considerably, leading to a tendency to increase the effect of mass transport resistance.

Cyclic voltamogram data obtained from Fig.3 are listed in Table 3. It can be seen that $I_{pa}/I_{pc}$ is as high as 1.547 at the VOSO$_4$ concentration of 0.3 mol·L$^{-1}$. This is possibly because V(V) ions obtained by oxidizing V(IV) ions are more likely to diffuse into the bulk solution in low concentration of VOSO$_4$ aqueous solution, resulting in a reduction of V(V) ions on electrode surfaces. Thus, the anodic peak current is much higher than the cathodic peak current. As the concentration of VOSO$_4$ increases, $I_{pa}/I_{pc}$ is reduced. When the concentration of VOSO$_4$ exceeds 1.0 mol·L$^{-1}$, $I_{pa}/I_{pc}$ is stabilized at more or less than 1.1. Also, the lowest potential difference ($\Delta V_p$) can be acquired with VOSO$_4$ of 1.5 mol·L$^{-1}$. When the concentration of VOSO$_4$ increases to be above 2.5 mol·L$^{-1}$, $\Delta V_p$ rapidly becomes larger, indicating the poor reversibility. The reason is that a remarkable increase in the viscosity of solution with its concentration leads to high mass transportability and a decrease in reversibility of the system as shown in Fig.4.

The polarization resistance ($R_p$) of the electrode reaction in 3 mol·L$^{-1}$ H$_2$SO$_4$ containing 0.3–3.0 mol·L$^{-1}$ VOSO$_4$ obtained from the linear polarization plot at a low sweep rate of 0.5 mV·s$^{-1}$ is shown in Fig.5. Two observations can be made from the data. Firstly, the polarization resistance increases sharply at concentration of VOSO$_4$ higher than 2.0 mol·L$^{-1}$ or lower than 0.5 mol·L$^{-1}$. Secondly, the polarization resistance is the lowest when the concentration of VOSO$_4$ is 1.0 mol·L$^{-1}$ with a slight increase when the concentration of VOSO$_4$ is between 1.5 and 2.0 mol·L$^{-1}$.

### Table 3 Cyclic voltamogram data of 0.3–3.0 mol·L$^{-1}$ VOSO$_4$ on a graphite electrode in 3 mol·L$^{-1}$ H$_2$SO$_4$ solution

<table>
<thead>
<tr>
<th>$c$(VOSO$_4$) (mol·L$^{-1}$)</th>
<th>$\Delta V_p$ [V]</th>
<th>$I_{pa}$/$I_{pc}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.3</td>
<td>0.182</td>
<td>1.547</td>
</tr>
<tr>
<td>0.5</td>
<td>0.163</td>
<td>1.350</td>
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<td>1.0</td>
<td>0.157</td>
<td>1.190</td>
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<tr>
<td>1.5</td>
<td>0.124</td>
<td>1.179</td>
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<tr>
<td>2.0</td>
<td>0.149</td>
<td>1.121</td>
</tr>
<tr>
<td>2.5</td>
<td>0.265</td>
<td>1.124</td>
</tr>
<tr>
<td>3.0</td>
<td>0.251</td>
<td>1.094</td>
</tr>
</tbody>
</table>

[a] $\Delta V_p$ means the difference between the anodic potential and the cathodic potential.

[b] $I_{pa}/I_{pc}$ means the ratio of the anodic peak current to the cathodic peak current.

### 2.3 Studies on the electrode process of concentrated V(IV)/V(V) by AC impedance

In the above three-electrode system, the electrode process of V(IV)/V(V) at high concentration is further investigated and verified below.

Fig.6 presents the AC impedance plot for 2.0 mol·L$^{-1}$ VOSO$_4$ in 1–4 mol·L$^{-1}$ H$_2$SO$_4$. It can be seen that the electrochemical polarization loop at high frequency and a slope line of 45° at medium or low frequency are observed when the H$_2$SO$_4$ concentration is 1 mol·L$^{-1}$, showing a high impedance. It indicates that the electrode process is controlled by both diffusion and electrochemical polarization. With the increase in concentration of H$_2$SO$_4$, the electrode process of the system turns to be controlled by diffusion with the lowest resistance when applying the 3 mol·L$^{-1}$ H$_2$SO$_4$. 

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Fig.4  Dependence of the viscosity of the V(IV)-3 mol·L$^{-1}$ H$_2$SO$_4$ solution on the VOSO$_4$ concentration at 20 °C

Fig.5  Polarization resistance vs the VOSO$_4$ concentration plots for the V(IV)-3 mol·L$^{-1}$ H$_2$SO$_4$ solution
From the above analysis, the equivalent circuit of the AC impedance spectra is shown in Fig. 7, where $R_L$ stands for Ohmic resistance, the plat double-layer capacitance ($C_{dl}$) is connected in parallel with the reaction resistance ($R_f$) to stand for the half-circle of electrochemical polarization at high frequency, $W_o$ stands for the Warburg diffusion resistance. Data obtained by simulating various components of the equivalent circuit are listed in Table 4. It can be seen that for 2.0 mol·L$^{-1}$ VOSO$_4$ in 1 mol·L$^{-1}$ H$_2$SO$_4$, the $R_f$ is the highest due to a small $n$(H$^+$) and the $R_L$ is the lowest when using 3 mol·L$^{-1}$ H$_2$SO$_4$. The $R_L$ depends mainly on the conductivity of electrode materials and electrolyte and contact resistance, wherein the $R_L$ directly reflects the differences from the conductivity of electrolytes. An increase in concentration of H$_2$SO$_4$ facilitates an improvement of the solution conductivity. However, as mentioned above, there is a sharp increase in the viscosity of solution when the concentration of H$_2$SO$_4$ is up to 4 mol·L$^{-1}$, so that the $R_L$ for the V(IV)-solution in 4 mol·L$^{-1}$ H$_2$SO$_4$ is higher than that for the V(IV)-solution in 3 mol·L$^{-1}$ H$_2$SO$_4$. It further indicates from the impedance investigation that for the 2.0 mol·L$^{-1}$ V(IV)-solution, 3 mol·L$^{-1}$ H$_2$SO$_4$ is favorable to be used as a supporting electrolyte.

**Table 4**  Equivalent circuit parameters for the AC impedance spectra of 2.0 mol·L$^{-1}$ VOSO$_4$ in 1–4 mol·L$^{-1}$ H$_2$SO$_4$

<table>
<thead>
<tr>
<th>$c$(H$_2$SO$_4$) (mol·L$^{-1}$)</th>
<th>$R_L$</th>
<th>$R_f$</th>
<th>$Z_c$</th>
<th>$W_o$-R</th>
<th>$W_o$-T</th>
<th>$W_o$-P</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8.069</td>
<td>25.85</td>
<td>1.74×10$^{-4}$</td>
<td>1.98</td>
<td>1.7×10$^{-5}$</td>
<td>0.22051</td>
</tr>
<tr>
<td>2</td>
<td>7.864</td>
<td>1.924</td>
<td>3.55×10$^{-4}$</td>
<td>204</td>
<td>2.445</td>
<td>0.42337</td>
</tr>
<tr>
<td>3</td>
<td>5.554</td>
<td>2.425</td>
<td>0.0224</td>
<td>3.98</td>
<td>0.0130</td>
<td>0.25865</td>
</tr>
<tr>
<td>4</td>
<td>7.276</td>
<td>1.001</td>
<td>5.56×10$^{-5}$</td>
<td>3.76</td>
<td>7.2×10$^{-3}$</td>
<td>0.26660</td>
</tr>
</tbody>
</table>

$R_L$, $R_f$ are the same as Fig. 7; $Z_c$: capacitor resistance; $W_o$-R, $W_o$-T, and $W_o$-P are three Parameters of the Warburg resistance.
controlled by diffusion, whereas the impedance is the smallest when the concentration of VOSO_4 is 2.0 mol·L⁻¹, slightly larger for 1.5 mol·L⁻¹ VOSO_4.

Components parameters of the equivalent circuit obtained by simulating the impedance plot in Fig.7 are listed in Table 5. It can be seen that as the concentration of VOSO_4 is raised, the \( R_1 \) increases step by step, especially showing a marked increase for 3.0 mol·L⁻¹ VOSO_4. It is because of the fact that the conductivity of the VOSO_4 solution is mainly dependent on H₂SO₄ due to existence of V(IV) as a complex ion with a large diameter. It is known from the above statements that in 3 mol·L⁻¹ H₂SO₄, the viscosity of the solution continues to increase as well as the \( R_1 \) with an enhancement in the concentration of VOSO_4. In particular, when the concentration of VOSO_4 is up to 3 mol·L⁻¹, the viscosity of the solution increases considerably as well as the \( R_1 \). Thus, from the viewpoint of the electrode process kinetics and giving attention to the energy density of batteries, the preferable concentration of VOSO_4 ranges from 1.5 to 2.0 mol·L⁻¹.

3 Conclusion

Applying the cyclic voltammetry, linear sweep voltammetry at a low scan rate and impedance technique, the effects of the concentration of V(IV) and H₂SO₄ on the electrode process were investigated in the range of relatively high concentration. It is shown that for the V(IV) solution of 2.0 mol·L⁻¹, when using H₂SO₄ below 2 mol·L⁻¹ as the supporting electrolyte, the polarization for the electrode reaction of V(IV)/V(V) is large with a poor reversibility and the electrode process is controlled by the electrochemical polarization and diffusion, together. When using H₂SO₄ above 2 mol·L⁻¹, the reversibility of the V(IV)/V(V) reaction is significantly enhanced and the electrode process is controlled by diffusion with a decrease in impedance as the concentration of H₂SO₄ is raised. Whereas, when the concentration of H₂SO₄ is up to 4 mol·L⁻¹, the high viscosity of solution and the high mass transportation resistance result in a slight increase in impedance. In 3 mol·L⁻¹ H₂SO₄, with the concentration of V(IV) increasing, the reversibility and kinetics of the system is improved with a decrease in impedance. However, when the concentration of V(IV) is beyond 2.0 mol·L⁻¹, the viscosity of solution is so high that the mass transportation resistance would increase remarkably, leading to a deterioration in the electrochemical performance of V(IV)/V(V) with an increase in impedance. Therefore, considering comprehensively two factors of the electrode reaction kinetics and energy density of batteries, the optimal concentration of the V(IV) solution is 1.5–2.0 mol·L⁻¹ in 3 mol·L⁻¹ H₂SO₄.

References

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Table 5 Equivalent circuit parameters for the AC impedance spectra of 1.0–3.0 mol·L⁻¹ VOSO_4 in 3 mol·L⁻¹ H₂SO₄

<table>
<thead>
<tr>
<th>c (VOSO_4) (mol·L⁻¹)</th>
<th>( R_1 ) (Ω·cm²)</th>
<th>( R_2 ) (Ω·cm²)</th>
<th>( Z_c ) (Ω·cm²)</th>
<th>Wo-R</th>
<th>Wo-T</th>
<th>Wo-P</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.0</td>
<td>3.826</td>
<td>0.4876</td>
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<td>1.5</td>
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<td>2.425</td>
<td>0.022400</td>
<td>3.980</td>
<td>0.01300</td>
<td>0.25865</td>
</tr>
<tr>
<td>3.0</td>
<td>14.38</td>
<td>1.537</td>
<td>0.055498</td>
<td>0.05599</td>
<td>1.159×10⁶</td>
<td>0.27779</td>
</tr>
</tbody>
</table>

\( R_1, R_2, \) and \( Z_c \) are the same as Table 4.