Enhanced Energy Storage Performance of Sodium Rhodizonate: Investigating the Role of Potassium Chloride and Sodium Sulfate Doping

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**Abstract.** The necessity of new renewable energy storage is high since the energy demand is high among the human population. With the emerging market, *the recyclability of the materials is an essential consideration since the waste of after-used material for energy storage will be hazardous to the environment, or the high cost of recycling will lower the economic scale of the materials*. Sodium Rhodizonate (SR) is a highly considerable material for energy storage since the theoretical energy storage is high, along with the abundance of sodium and the excellent sustainability of the environment. The characteristics of SR as an organic compound pose an opportunity to be eco-friendly. The main challenge for SR is the performance of storing energy and the stability of the material to pursue a high number of cycles. *The general goal of our research is to focus on water-based SR and various doping materials to improve energy storage performance or specific capacity.* The result showed that the performance *of the Specific Capacity* of SR with doping sodium sulfate increases with the optimum improvement to 62% compared to pure SR, while the performance of SR with doping potassium chloride increases by nearly double the specific capacity (~ 98%) compared to pure SR. However, the performance stability of the SR and the composites falls by a significant number (~95%) with the doping. The decomposition of the material is the main reason for the substantial fall in the performance. *Furthermore, the milestone of our goal is to improve the performance of SR has achieved by doping the material.*

# INTRODUCTION

The rise of the earth’s surface temperature until 4oC in 2100 [1] has alerted all researchers worldwide to overcome the situation since it has caused the sea level to rise more than ever before. Global leaders have committed to fighting against the rising temperature through the Paris Agreement to keep the global temperature from exceeding 1.5 oC by reducing emissions to 45% by 2023 and reaching Net Zero Emission by 2050 [2]. The shift towards attaining a net-zero global ecosystem is one of humanity's paramount hurdles. It mandates a sweeping transformation in production, consumption, and mobility approaches. Currently, the energy domain serves as the primary contributor, accounting for roughly three-quarters of greenhouse gas emissions, thereby holding critical importance in circumventing the severe repercussions of climate change. Substituting detrimental coal, gas, and oil-driven energy production with sustainable alternatives such as wind or solar power presents a substantial avenue for significantly curbing carbon emissions.

Until 2022, oil still possesses the highest fuel consumption globally, at 32%, which dominates the total source of global consumption [3] (Fig 1a). Furthermore, the most dominant sector that consumes oil is road, which consists of vehicles commonly using fossil fuel as a fuel source or named an Internal Combustion Engine (ICE) [4] (Fig 1b). One of the effects of massively consuming oil as an energy source is CO2 emission since the product side of incomplete combustion reaction [5], which is related to the Greenhouse Effect.

The transportation sector accounted for 20.7% of total global CO2 emissions during 2022 [6] contributing to the cars and vans subsector by 48% [7]. Furthermore, to avoid CO2 being released into the atmosphere, researchers need to find alternatives besides oil as an energy resource.



(1a) (1b)

Figure 1a. Global primary energy consumption in 2022 is categorized by fuel type; 1(b) Oil demand share by OECD (Organisation for Economic Cooperation and Development) in 2022 classified by sectors.



(2a) (2b)

Figure 2a. Sectors categorize global CO2 emission in 2022; Figure 2b. The breakdown of CO2 emission in transportation in 2022 is classified by sub-sectors.

Future research will likely create cleaner energy resources to replace oil as a main fuel resource to reduce CO2 emissions since cars and vans are the most significant producers of CO2 released into the atmosphere, including the road as a top sector for consuming oil. Hence, it makes sense for the researchers to find another alternative fuel resource for cars and vans.

Electrification of vehicles is one of the global action plans from the automotive industry; this route has been taken due to the ability of electricity to be a fuel resource for vehicles without emitting any hazardous gases into the atmosphere [8]. Electric vehicles (EVs) are gaining advantages in terms of low emission over ICE and performance since the EV will reach the maximum torque from the beginning [9]. Furthermore, the running cost for EVs also outperformed that of ICE vehicles [10].

One of the main challenges of EV implementation in replacing oil is related to the development of batteries since they play a critical role in preserving energy. The development of batteries was started in 1800 by Alessandro Volta with the “Voltaic Pile” mechanism using stacked discs made of silver, copper, or brass [11]. The Battery Energy Storage System (BESS) is facing some areas for improvement before completely replacing the oil due to its life span, operating temperature, depth of discharge, self-discharge, efficiency, and energy density [12].

Lithium has become the most implemented material for battery construction because of its high energy, power density, long life span, and environmental friendliness [13] also called lithium-ion batteries (LIBs). In 2021, the global consumption of lithium showed that the battery industry consumes up to 72%, among other sectors [14]. However, the abundance of lithium is relatively low for future demand, and it causes some upcoming problems, such as price issues due to a lack of supply in the market for long-term usage [15]. Hence, it makes some researchers figure out an alternative active material besides lithium for battery use.

Sodium-ion Batteries (SIBs) are widely known as an emerging new active material to replace lithium because its fundamental aspects, structure, valence electron, and mechanism of storing charge remain consistent, with the only variation being the substitution of lithium ions with sodium ions[16]. However, the development of SIBs to compete with the LIB’s performance is still ongoing since the number of SIBs publications is getting the hype this last decade.

One of the main handicaps of SIBs compared to LIBs is energy density due to their structure [17]. Sodium has more electrons than lithium; sodium has more electron shells, making sodium's diameter (1.02 Å) larger than lithium (0.76 Å). This situation leads to phase instability, transport properties, and interphase transformation [18]. The bigger ions make the capability to be stored (sterically) in the structure of an anode or cathode less likely. Hence, the researcher's primary purpose is to improve the performance of the SIB active material to compete with the LIB’s performance.

*Organic-based cathodes are known to be utilized for SIBs since their characteristics such as high tunable properties, relatively affordable, bio-degradable, and free from metal components [19]. In addition, Terpenoid organic compounds have drawn attention in the research of cathode materials for secondary batteries due to their superior theoretical capacity and energy density compared to other organic cathode materials [20].*

Sodium rhodizonate (SR) or Na2C6O6 is one of the ideal candidates for active material from organic-based family because of its high theoretical capacity of 250 mAh g-1 [21]; another advantage is gained from its C6O6 structure, where the layered structure is ideal for ion storage in an energy storage mechanism. Since the SR is classified as an organic compound, which has an excellent chance for sustainability because the waste is easy to handle. However, in the actual situation, the performance of SR is still lower than the theoretical capacity, which might have happened due to the irreversible phase during the charge-discharge cycle and unstable material physically and/or chemically during the charge-discharge cycle [22]. Researchers have successfully conducted various improvements by varying the binder [22], electrolyte [23] and compositing with the polyelectrolyte [24].

Our work focuses on a deep understanding of the raw performance of SR without any complex modification. The water-based SR form was also chosen to understand the behavior of SR when storing energy in that form since limited information has been published related to this condition. The SR will be doped with two families of doping from sulfate and chloride derivatives.

# EXPERIMENT

## Preparation of Water-based Sodium Rhodizonate

Water-based SR is prepared by dissolving it with a solvent that exhibits similar polarity. A certain amount of SR was added to obtain 0.6M within the solvent at 80oC. The dissolved SR will be placed at room temperature for 2 hours.

## Preparation of Water-based Sodium Rhodizonate Doped Potassium Chloride

Water-based SR-doped potassium chloride (KCl) (SR-PC) is made with the crystal forms for both substances. The SR-KS is prepared by dissolving it with a water-based solvent that exhibits similar polarity*. 0.167 grams of SR and potassium chloride were added to the solvent at 80oC. The variation of potassium chloride will be wt% 5%, 10%, 15%, 20%, and 25% labeled as SR-PC5, SR-PC10, SR-PC15, SR-PC20, and SR-PC25. The solution is gently stirred at 500 rpm for 15 minutes. The dissolved SR-PC will be placed at room temperature for 2 hours.*

## Preparation of Water-based Sodium Rhodizonate Doped Sodium Sulfate

Water-based SR-doped sodium sulfate (Na2SO4) (SR-SS) is made with the crystal forms for both substances. The SR-SS is prepared by dissolving it with a solvent that exhibits similar polarity. *0.167 grams of SR and sodium sulfate were added to the solvent at 80oC. The variation of sodium sulfate will be wt% 5%, 10%, 15%, 20%, and 25% labeled as a SR-SS5, SR-SS10; SR-SS15; SR-SS20; SR-SS25. The solution is gently stirred at 500 rpm for 15 minutes. The dissolved SR-SS will be placed at room temperature for 2 hours.*

## Measurement of the Performance

The electrochemical performances were carried out by the Cyclic Voltammetry (CV) and Galvanic Charge-Discharge (GCD) with the Corrtest CS250M to evaluate the storage mechanism and performance. GCD measurements were conducted with the condition that the material will be charged when the potential is lower than 0.5V; and discharged when the potential (V) is more than 1.5V. The CV measurement is carried out with the window current 0.5-1.5 mA.

# RESULT and discussion

## Pure Sodium Rhodizonate (SR)

This experiment concludes that SR can serve as an energy storage active material even in its simplest form. The GCD measurement result

To better understand the behavior of the active materials in stabler conditions, we will look at the trend after the second cycle to the fifteenth due to a more gradual degradation to understand material stability better, as shown in picture 3b. The performance trend of storing the energy (charging) of SR is decreased as a time function is near the power trend line by following the equation:

(1)

Q = Specific Capacity (mAh g-1)

*k*  = Material Stability Coefficient

x = Time (s)

*n* = Power or Exponent

Since the data trend follows the power trendline, we can generally perceive that the SR naturally has a massive degradation performance initially but will be more stable in the following cycles. *The k value gives us information about the performance of the battery in the initial period of usage because the k value will play an important role in the equation at the beginning of the series of x values; k value is the intrinsic value that emerges for each combination of materials depend on the interactions between compounds. The k value quantifies material stability at the beginning of the reaction, which reflects the degradation in the early stages*. Furthermore, the *n* value will play an important role in the degradation rate of energy storage performance as a time function, as a smaller *n* value will indicate the degradation rate of the battery will be more consistent throughout the time. Still, a higher *n* value leads to the more degradation rate at the beginning of usage, but the rate will decrease throughout the time. The most desired combination for the battery performance will be a low *k* value and a high *n* value, resulting in energy storage with low initial degradation and a more gradual decrease in the following cycles.

3a

3b

3b

Figure 3a. The Charging Specific Capacity of Sodium Rhodizonate; Figure 3b. The degradation rate of sodium rhodizonate until 15th cycle

The peak performance storing energy of SR is ± 188 mAh/g with the material stability factor around 298 (Figure 3a). The phenomenon is also supported by the period of charging and discharging. Table 1 shows that the charging time for the first cycle is extremely high compared to the following cycles, which is correlated with the capacity of the first cycle result. The short discharging time leads to the unstable ions retained within the structure of active materials.

TABLE 1 Comparison of charging and discharging time of sodium rhodizonate

|  |  |  |
| --- | --- | --- |
| **Cycle** | **Charging Time (s)** | **Discharging Time (s)** |
| 1 | 966.18 | 0.16 |
| 2 | 23.42 | 0.14 |
| 3 | 20.30 | 0.12 |
| 4 | 16.00 | 0.02 |
| 5 | 10.40 | 0.09 |

## Sodium Rhodizonate doped Potassium Chloride (SR-PC)

The effort to improve the performance of SR can be achieved by adding the doping agent to enhance the ions within the composites. In this experiment, potassium chloride was added to the sodium rhodizonate with various concentrations to understand the effect of the doping presence. Potassium chloride is a salt doping agent with pH neutral; it dissociates the anion, and the cation will be separated completely within the solvent to maximize the number of ions within the composites. However, the presence of potassium chloride is related to increasing the specific capacity of the active materials instead of the stability of the chemical and mechanical properties.

The result showed that potassium chloride in the sodium rhodizonate has successfully improved the peak performance of storage throughout the variations (Figure 4a). The presence of potassium ions playing an important role to reduce the charge transfer resistance [25], the appropriate concentration of potassium can enlarge the interlayer spacing of the material, thereby aiding the diffusion of sodium ions [26].

The highest peak performance is achieved with the 15 wt% of potassium chloride by ~81% from the original SR, whilst the least improvement is achieved by the 25 wt% of potassium chloride with 46%. However, the ability to retain the chemical and mechanical stability of composites during charge-discharge is severe and needs further improvement (Figure 4b). The CV measurement (Figure 5f) confirmed the reason behind the impairment of the storage performance related to the depletion of the ions moving through electrodes since the response voltage from the active material is weakened through the subsequent cycles.

4a

4b

Figure 4(a) The peak performance of SR-PC15 contributes the most effective to enlarge the interlayer spacing material; Figure 4(b) The charging result of SR-PC15 (the optimum condition) until the 50th cycle.

Taking further the detail of the result, to understand the behavior of chemical and mechanical stability, we will deep look and analyze the trend after the second cycle until the fifteenth; we choose until the fifteenth cycle due to the following cycle being very flat, which leads to a better condition for the retainment, but we need to consider the least desirable condition (Figure 5a-5e).

The *n* value for all variations shows a greater value than the original SR, displaying the opportunity for a more sustainable degradation rate in the next cycles, leading to the better performance of active materials when deployed as energy storage material. The optimum condition is achieved with the 20wt% rather than the original SR (Table 2).

On the contrary, the *k* value of all variations shows fewer solid results, which leads to the materials having a huge degradation rate in the early usage unless the 25wt%. The *k* value performance of the SR-PC25 is inseparable from the fact that the degradation from the second cycle to the fifteenth is fewer among the others. In fact, the performance of composites is not only dependent on the *k* value but also their interaction with the other parameters.

The R-square value of the SR-PC composites showed that the materials are more likely to follow the power trendline; this implies that the doping makes the composites more reliable in the long term since the degradation rate will be more stable in the following cycles rather than the original SR.

TABLE 2 The n, k, and r-square values of the SR-PC composites

|  |  |  |  |
| --- | --- | --- | --- |
| Cycle | n | k |  |
| SR | 1.206 | 298 | 0.7932 |
| SR-PC5 | 1.536 | 1718.1 | 0.9471 |
| SR-PC10 | 1.536 | 7845.7 | 0.9293 |
| SR-PC15 | 1.421 | 1456 | 0.9698 |
| SR-PC20 | 1.61 | 3243.6 | 0.9821 |
| SR-PC25 | 1.361 | 256.1 | 0.9415 |

5a

5b

5c

5d

5e

5f

Figure 5(a-e) the degradation rate of SR-PC5, SR-PC10. SR-PC15, SR-PC20, and SR-PC25. Figure 5f the CV measurement 0.5 - 1.5 mA SR-PC15, the tendency for the lower potential range indicates the lesser mechanical stability.

The efficiency of the storage performance of the composites can be evaluated by projecting the degradation rate of each composite using the equations (Table 3). The projection will consider the charging time until 100s, then calculate the average of each result. The ΔQ data is the calculation degradation rate of the recent cycles with the following cycles, resulting in a percentage representing the deterioration in the subsequent cycles. The result showed that all the composites had a slightly inefficient performance (>25%) when compared with the original SR; it makes the dopant potassium chloride the ideal doping for the SR to enhance the overall storage performance, but the retainment chemical and mechanical stability is slightly lower with the original SR (Table 4).

TABLE 3 The projection of degradation rate for all SR-PC composites until 100s

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| x (s) | SR | SR-NS5 | SR-NS10 | SR-NS15 | SR-NS20 | SR-NS25 |
| 10 | 18.54 | 50.01 | 228.37 | 55.23 | 79.62 | 11.15 |
| 20 | 8.04 | 17.25 | 78.75 | 20.63 | 26.08 | 4.34 |
| 30 | 4.93 | 9.25 | 42.24 | 11.59 | 13.58 | 2.50 |
| 40 | 3.48 | 5.95 | 27.16 | 7.70 | 8.54 | 1.69 |
| 50 | 2.66 | 4.22 | 19.28 | 5.61 | 5.97 | 1.25 |
| 60 | 2.14 | 3.19 | 14.57 | 4.33 | 4.45 | 0.97 |
| 70 | 1.77 | 2.52 | 11.50 | 3.48 | 3.47 | 0.79 |
| 80 | 1.51 | 2.05 | 9.36 | 2.88 | 2.80 | 0.66 |
| 90 | 1.31 | 1.71 | 7.81 | 2.43 | 2.32 | 0.56 |
| 100 | 1.15 | 1.46 | 6.65 | 2.09 | 1.95 | 0.49 |

TABLE 4 The degradation rate of storing energy calculation for the SR-PC composites

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| ΔQ | SR | SR-PC5 | SR-PC10 | SR-PC15 | SR-PC20 | SR-PC25 |
| 10 – 20 | 57% | 66% | 66% | 63% | 67% | 61% |
| 20 – 30 | 39% | 46% | 46% | 44% | 48% | 42% |
| 30 – 40 | 29% | 36% | 36% | 34% | 37% | 32% |
| 40 – 50 | 24% | 29% | 29% | 27% | 30% | 26% |
| 50 – 60 | 20% | 24% | 24% | 23% | 25% | 22% |
| 60 – 70 | 17% | 21% | 21% | 20% | 22% | 19% |
| 70 – 80 | 15% | 19% | 19% | 17% | 19% | 17% |
| 80 – 90 | 13% | 17% | 17% | 15% | 17% | 15% |
| 90 – 100 | 12% | 15% | 15% | 14% | 16% | 13% |
| Average | 25% | 30% | 30% | 28% | 31% | 28% |

## Sodium Rhodizonate doped Sodium Sulfate (SR-NS)

Another neutral salt, sodium sulfate, has been deployed as a dopant for the SR to understand the effect of sodium ions in the composite. The main idea is to utilize sodium sulfate to obtain additional sodium ions as active ions in accordance with the respective improvement of storage ability. The result (Figure 6a) showed that doping with the sodium sulfate improved the storage performance of SR with the optimum condition at 10 wt% (SR-NS10); the storage performance continues to experience a declining trend throughout the increasing sodium sulfate concentration. The proposed explanation is that adding sodium ions improves the storage performance due to more active ions moving through the electrodes, but after the optimum condition is achieved, the steric hindrance will play an important role in causing the storing performance of SR-NS composites to decrease. The improvement for storage performance may come from the larger electronic conductivity[27]]. However, the performance of SR-NS is facing a similar problem with the other composites, which is severe with the chemical and mechanical stability in causing the massive degradation of the energy storage performance with the following cycles (picture 6b). From the CV measurement, it clearly can be seen that the ability to storage the energy is reduced in the following cycles. The response potential from the active materials is getting lower, which indicates that the structure is deformed in subsequent cycles.

6a

6b

Figure 6a The peak performance of SR-NS10 was contributed by the existence of sodium ions that enhance the moving ions within the materials; Figure 6b The charging result of SR-NS10 (the optimum condition) until the 50th cycle.

Besides the peak performance, the storage performance of the SR-NS composites also surpasses the SR. The data from the second until the fifteenth cycles showed that the behavior of SR-NS composites became more suitable with the model power curve, making the active materials more predictable, which was indicated by the R-square values near to ideal instead of SR (Figure 7a-7e). Furthermore, the ability to maintain the energy storage capacity (*n* value) is generally improved from the original SR, but the degradation in the early cycles seems to decline, which can be seen by the escalating of *k* values (Table 5).

7a

7b

7c

7d

7e

Figure 7(a-e) The degradation rate of SR-NS5, SR-NS10. SR-NS15, SR-NS20, and SR-NS25. Figure 7f the CV measurement 0.5 - 1.5 mA SR-NS10

To calculate the efficiency of the storage performance, we simulate the equation from each composite by 10 to 100 seconds (Table 6), resulting in the percentage of degradation from the composites (Table 7). From the calculation, it is visible that only the SR-NS15 achieves a better performance efficiency energy storage (22%) than the original SR (25). In conclusion, the performance of SR-NS composites is improved compared to the original SR. The level of stored energy is higher than the original SR and is reflected in the actual specific energy and the *k* value, representing the energy level value. However, most SR-NS composites face a higher overall degradation rate unless SR-NS15.

TABLE 5 The n, k, and r-square values of the SR-NS composites

|  |  |  |  |
| --- | --- | --- | --- |
| **Cycle** | **k** | **n** |  |
| SR | 298 | 1.206 | 0.7932 |
| SR-NS5 | 7876 | 1.796 | 0.9749 |
| SR-NS10 | 829 | 1.407 | 0.9548 |
| SR-NS15 | 115.65 | 1.045 | 0.9564 |
| SR-NS20 | 681.5 | 1.452 | 0.9248 |
| SR-NS25 | 8410.6 | 1.763 | 0.9411 |

TABLE 6 The projection of degradation rate for all SR-NS composites until 100s

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| x(s) | SR | SR-NS5 | SR-NS10 | SR-NS15 | SR-NS20 | SR-NS25 |
| 10 | 18.54 | 125.98 | 32.48 | 10.43 | 24.07 | 145.15 |
| 20 | 8.04 | 36.28 | 12.25 | 5.05 | 8.80 | 42.77 |
| 30 | 4.93 | 17.51 | 6.92 | 3.31 | 4.88 | 20.92 |
| 40 | 3.48 | 10.45 | 4.62 | 2.45 | 3.22 | 12.60 |
| 50 | 2.66 | 7.00 | 3.37 | 1.94 | 2.33 | 8.50 |
| 60 | 2.14 | 5.04 | 2.61 | 1.60 | 1.78 | 6.17 |
| 70 | 1.77 | 3.82 | 2.10 | 1.36 | 1.43 | 4.70 |
| 80 | 1.51 | 3.01 | 1.74 | 1.19 | 1.18 | 3.71 |
| 90 | 1.31 | 2.43 | 1.48 | 1.05 | 0.99 | 3.02 |
| 100 | 1.15 | 2.02 | 1.27 | 0.94 | 0.85 | 2.51 |

TABLE 7 The degradation rate of storing energy calculation for the SR-NS composites

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Δx (s) | SR | SR-NS5 | SR-NS10 | SR-NS15 | SR-NS20 | SR-NS25 |
| 10 – 20 | 57% | 71% | 62% | 52% | 63% | 71% |
| 20 – 30 | 39% | 52% | 43% | 35% | 44% | 51% |
| 30 – 40 | 29% | 40% | 33% | 26% | 34% | 40% |
| 40 – 50 | 24% | 33% | 27% | 21% | 28% | 33% |
| 50 – 60 | 20% | 28% | 23% | 17% | 23% | 27% |
| 60 – 70 | 17% | 24% | 19% | 15% | 20% | 24% |
| 70 – 80 | 15% | 21% | 17% | 13% | 18% | 21% |
| 80 – 90 | 13% | 19% | 15% | 12% | 16% | 19% |
| 90 – 100 | 12% | 17% | 14% | 10% | 14% | 17% |
| Average | 25% | 34% | 28% | 22% | 29% | 34% |

# conclusion

The performance of SR has successfully improved with the presence of potassium chloride and sodium sulfate. The performance of SR-PC composites achieved a better result than the SR-NS, which is the possible main reason that the saturation of sodium ions makes the reaction inefficient. The next step of these findings is related to improving the chemical and mechanical stability to improve the possibility of utilizing these active materials for energy storage. Compositing the materials with the binder or other materials such as Polyaniline with cross-linkage characteristics will be a robust future improvement also improve the specific capacity since the polyaniline is a conductive polymer. The potential of industrialization of this material can be robust since the composites were built by one-hundred percent organic compound, leading to the eco-friendly material.

# acknowledgments

I want to thank the Indonesia Endowment Fund for Education (LPDP) with registered number 0004804/IPA/D/2/lpdp2022 from the Ministry of Finance Republic Indonesia for granting the scholarship and supporting this research.

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