

## **Impact of Heteroatoms and Transition Metal Co-doping on the performance of Graphene based Supercapacitor electrodes**

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### **1. ABSTRACT**

The escalating demand for clean and sustainable energy sources in the face of depleting non-renewable resources has spurred extensive research into energy storage technologies. Supercapacitors, with their high specific power, rapid charge-discharge cycles, and extended lifecycle, emerge as promising candidates to bridge the gap between batteries and conventional capacitors. However, their inherent limitation of low energy density prompts investigations into enhancing their performance. This review focuses on the pivotal role of heteroatom co-doping, specifically nitrogen (N), boron (B), phosphorus (P), sulphur (S), and transition metals (TM), in graphene-based supercapacitors. Examining the effects of co-doping on graphene's structural stability, density of states, quantum capacitance, and surface charge density, the study reveals that the incorporation of multiple dopants creates synergistic effects, significantly improving the energy storage capabilities of supercapacitors. Notably, the co-doping of TMN<sub>x</sub> (TM = Ti, V, Cr, Mn, Co, Ni) demonstrates remarkable potential, enhancing quantum capacitance and offering design strategies for high-performance graphene-based supercapacitors. The findings contribute valuable insights into the development of efficient electrode materials for advanced energy storage applications.

### **2. INTRODUCTION**

Continuous depletion of non-renewable energy resources and the increasing of environmental pollution, has severely affected the world ecology as well as the economy in the present day. The solution is to utilise renewable energy resources for power generation. But one main issue in renewable power sources is that they are not continuous and homogenous with time. Therefore, it necessitates the urgent pursuit of research to develop and deliver new energy storage devices that can provide renewable, green and clean energy for mankind. Among the renewable energy storage devices, supercapacitors are standing out as a potential breakthrough, as they possess impressive characteristics such as high specific power, fast charging-discharging cycles, a wide operating temperature range (can withstand from  $-40\text{ }^{\circ}\text{C}$  to  $70\text{ }^{\circ}\text{C}$ ), and long-life cycle compared to conventional capacitors and some battery types [1]. Unlike batteries, which store energy chemically, supercapacitors store energy electrostatically. Accordingly, rather of storing energy through chemical reactions, they store energy through the separation of charge. The formation of an electrical double layer at the interface between the electrode and the electrolyte is the primary mechanism underlying supercapacitors. Ions from the

electrolyte are drawn to the electrode surface when a voltage is applied across them, creating a double layer of charge. Supercapacitors are widely used in consumer electronics, memory back-up systems, industrial power and energy management, low-emission hybrid electric vehicles, and fuel cell vehicles [2]. In view of its advantages, supercapacitors can fill the gap between batteries and conventional capacitors. One type of supercapacitor, is known as electric double-layer capacitors (EDLCs), have a significantly higher capacitance than conventional dielectric capacitors. A major disadvantage of supercapacitors is their low energy density [3]. Essentially, the charge stored at the electrode-electrolyte interface acts as what determines the capacitance values of EDLCs. As the mechanism of EDLCs is a surface phenomenon, the electrolyte's accessible surface area has a major impact on how efficiently the charge-discharge process operates [4]. Thus, enhancement of capacitance requires a large surface area, strong electrical conductivity, high mechanical and chemical stability, and optimised nanostructure. In supercapacitors, quantum capacitance ( $Q_c$ ) plays a crucial role in understanding their exceptional charge storage ability.  $Q_c$  is an intrinsic property of the materials, which arises from the kinetic, exchange–correlation, and electron–phonon interaction energies in the total energy functional. It is a measure of the change in the number of electrons in a material's conduction band per unit change in the electrochemical potential. Graphene is a single layer of sp<sup>2</sup>-bonded carbon atoms, densely packed into a honeycomb structure. Nowadays, chemically modified graphene is the most promising carbon material for use in supercapacitors due to its unique characteristics, which include a large specific surface area, excellent stability high mobility of charge carriers ( $200,000\text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ ) and good electrical conductivity [5]. Nevertheless, the lack of catalytic behaviour and zero band gap significantly limit the practical implementations of pure graphene. Thus, efforts are being made to unlock the potential of graphene by transforming its structure to tune its physical and chemical properties.

Doping is an approach that effectively tunes the intrinsic characteristics and electronic structure of graphene by altering the chemical reactions occurring at the interfaces, leading to excellent electrochemical performance [6]. Research on heteroatom doping in graphene has been substantial, particularly for energy-related applications [7]. The transition metals include Ti, V, Cr, Mn, Co, and Ni. It is representative and theoretical to investigate how the doping of these elements affects the structure, stability, and quantum capacitance of graphene. Investigating whether TMN<sub>x</sub>-G (TM=Ti, V, Cr, Mn, Co, or Ni) can demonstrate significant potential as the electrode material of supercapacitors is therefore an extremely significant endeavour. The outcomes of this review will provide a foundation on the significance of co-doping heteroatoms and transition metals on developing and manufacturing graphene-based supercapacitors.

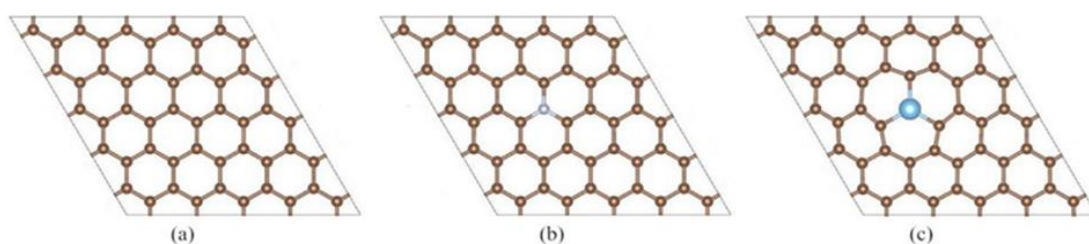


Fig. 1- Optimised structures of (a) perfect, (b) N-doped, and (c) Ti-doped graphene monolayers [16]

Capacitance of supercapacitors has been found to be efficiently enhanced by nitrogen doping among the heteroatoms [8-11]. It has also been demonstrated that the synergistic effects of co-doping two or more atom types result in even greater capacitances. Co-doping introduces dopants that may donate or receive electrons, increasing the charge carrier density in the material. The accumulation of charge carriers at the interface between a material and an electrolyte can result in an increase in the capacitance known as the EDLC capacitance. Additionally, co-doping can improve a material's surface area by introducing crystal lattice defects. This can lead to an increase in the pseudocapacitance, which is the capacitance that arises from the redox reactions of charge carriers on the surface of a material [12].

### 3. HETEROATOM CO-DOPED GRAPHENE

B, N, P, and S are considered to be the more suitable dopants for graphene due to their similarity to carbon atoms. Table 1 provides for a comparison of their electronegativities, valence electron counts, sizes, and electronic configurations. Included for comparison are the characteristics associated with carbon atoms. The elements that have been determined to be more appropriate as dopants for graphene include B, N, P, and S because of their resemblance to carbon atoms. The characteristics of the carbon atom are also included for comparison.

Table 1 - Electronic structure related data for different dopants for carbon [13]

Element	Van der Waals radius (pm)	Electronic configuration	No. of valence electrons	Electronegativity
B	180	$1s^2 2s^2 2p^1$	3	2.04
N	155	$1s^2 2s^2 2p^3$	5	3.04
P	195	$1s^2 2s^2 2p^6 3s^2 3p^3$	5	2.19
S	180	$1s^2 2s^2 2p^6 3s^2 3p^4$	6	2.58
C	170	$1s^2 2s^2 2p^2$	4	2.55

N is considered to be the most significant heteroatom dopant to enhance the capacitance characteristics of carbon materials as it adds more sites for faradic redox reactions. N-doping alters the electronic structure of graphene, improving charge carrier density in addition to the pseudocapacitive contribution. Thus, the creation of Nitrogen doped Graphene for supercapacitor electrodes has been the subject of several published investigations [14-18].

B also stands out with N as primary choices to enhance the supercapacitor properties of graphene owing to their strategic positioning around carbon in the periodic table. Their smaller and larger electronegativities compared to carbon make them particularly attractive for developing metal-free catalysts, capitalising on the charge imbalance they introduce. In 2013, Zheng et al. reported the successful preparation of graphene co-doped with B and N [19]. It has been applied for the fabrication of both symmetric and asymmetric supercapacitor electrodes showcased that, in an alkaline aqueous electrolyte, the specific capacitance reached  $283 \text{ F}\cdot\text{g}^{-1}$  at  $1 \text{ A}\cdot\text{g}^{-1}$ , surpassing the performance observed with graphene doped solely with N or B [20]. Another example is Mullen's group, who demonstrated that incorporating B/N heteroatoms into the three-dimensional structure of graphene aerogels (3D-BNGAs) significantly improved the performance of an all-solid-state supercapacitor. This improvement was attributed to the reduction in device thickness, resulting in increased energy density (approximately  $8.65 \text{ Wh}\cdot\text{kg}^{-1}$ ), higher specific capacitance (about  $62 \text{ F}\cdot\text{g}^{-1}$ ), and improved rate capability [20]. Additionally, B/N-graphene synthesised through thermal annealing of graphene oxide with melamine diborate at  $700^\circ\text{C}$  exhibited an enhanced capacitance of  $130.7 \text{ F}\cdot\text{g}^{-1}$ , surpassing that of singly doped and undoped graphene-based supercapacitors [21]. Chen et al. demonstrated that charge storage properties of B/N co-doped graphene-like carbons could be controlled [20]. The BNC-900 device exhibited high specific capacitance retention ( $\sim 78.2\%$ ), good cycling stability over 1000 cycles at  $100 \text{ A}\cdot\text{g}^{-1}$ , and an excellent specific capacitance of  $225 \text{ F}\cdot\text{g}^{-1}$  at  $0.25 \text{ A}\cdot\text{g}^{-1}$ . The enhanced electrical performance was attributed to the synergistic effect of B and N dopants, as well as the modified electronic and surface properties of the doped graphene nanostructure [21]. Oxygen and Nitrogen co-doping on Graphene also increase electroactive sites. O/N co-doping introduces heteroatoms with lone pair electrons and empty orbitals, respectively. Studies with N-doped graphene oxide (N-GO) and O/N-co-doped carbon nano walls (O/N-CNWs) demonstrated significant increases in specific capacitance compared to undoped counterparts according to Li et al., 2017 [22] and Wang et al., 2016 [23]. The combination of O and N dopants creates a synergistic effect, maximising the beneficial effects of each element. Co-doping graphene with both phosphorus (P) and nitrogen (N) also emerges as a potent strategy to boost supercapacitor performance. Phosphorus, with its larger atomic size, and nitrogen, with its electronegativity, also create a synergistic effect making P and N co-doped graphene a promising material for energy storage applications. The co-doping of graphene with sulphur and nitrogen has also garnered attention for its potential impact on supercapacitor performance. A high sulphur content

(3.36%) of nitrogen and sulphur co-doped nanoporous carbon was created by Chen et al. [24]; at a current density of 1 A/g, the material demonstrated a specific capacitance of 73 F/g.

#### 4. TRANSITION METAL CO-DOPED GRAPHENE

Transition metals possess unique *d*-orbitals that readily hybridise with the  $\pi$ -orbitals of graphene. This hybridization introduces new energy states near the Fermi level of graphene, significantly increasing the density of states (DOS) in this crucial region. A higher DOS translates to more available pathways for electron storage, directly boosting quantum capacitance. Doping with multiple transition metals (co-doping) can create synergistic effects, further enhancing quantum capacitance and other desirable properties. Heteroatom co-doping, combining transition metals with elements like N or B, opens up even more possibilities for optimising performance.

The maximum Quantum Capacitance ( $Q_c$ ) of the six constructions for transition-metal doped graphene, specifically TiG, VG, CrG, MnG, CoG, and NiG, is 147.56  $\mu\text{F}/\text{cm}^2$  at 0.24 V, 76.55  $\mu\text{F}/\text{cm}^2$  at 0.08 V, 173.49  $\mu\text{F}/\text{cm}^2$  at 0.13 V, 112.60  $\mu\text{F}/\text{cm}^2$  at 0.15 V, 165.49  $\mu\text{F}/\text{cm}^2$  at 0.24 V, and 239.28  $\mu\text{F}/\text{cm}^2$  at 0.19 V, respectively. It is discovered that NiG has the greatest  $Q_c$  value. Prior research indicated that co-doping with transition metals and N might provide a higher capacitance than doping with only one dopant [16,26].

#### 5. THE EFFECT OF TMN<sub>x</sub>-G CO-DOPING OF GRAPHENE

##### 5.1 Structure and stability

The calculated average TM-N bond lengths between dopant TM atom and its one-coordinate N atoms considering average distances are 2.10, 2.08, 2.09, 1.97, 1.93, 1.92 Å for TiN-G, VN-G, CrN-G, MnN-G, CoN-G and NiN-G respectively. Thus, for graphene doped with different TM, the average bond lengths of the TMN<sub>x</sub>-G are in the order: TiN<sub>x</sub>-G > CrN-G > VN-G > MnN-G > CoN-G > NiN-G [29]. Shorter bond lengths generally indicate stronger bonds and therefore a more stable structure. But bond length alone can be misleading in some cases as multiple bonds (double, triple) are shorter than single bonds, but not necessarily stronger. Additionally, bond lengths can be influenced by factors like steric hindrance and hybridization. Hence, bond length can be a good initial indicator, especially if multiple bonds are not present. The formation energies ( $E_f$ ) for above-mentioned co-doped structures are calculated and shown in Table 2 below. As shown, the values of  $E_f$  all are negative, which indicates the co-doping structures are in a relatively stable state. When the doping positions of the N atoms are the same, the  $E_f$  of the TMN<sub>x</sub>-G are in the order: CrN-G > MnN-G > VN-G > TiN-G > CoN-G > NiN-G. This order indicates that NiN<sub>x</sub> co-doped graphene is the most stable while CrN co-doped graphene is the least stable. Combining bond length with formation energy can provide a more complete picture of bond strength and stability. The values of  $E_f$  are consistent with the calculated TM-N bond lengths. For example, the NiN-G has the least  $E_f$  (-2.67 eV)

and the shortest TM-N bond length (1.92 Å) [25,28]. For the same dopant TM, the  $E_f$  of TMN<sub>x</sub>-G is in the order of TMN1-G > TMN2-G > TMN3-G > TMN4-G. This indicates that the stability of graphene improves with the increase of doping numbers of N atoms.

Table 2- Formation energies ( $E_f$ ), maximum quantum capacitance ( $Q_{c-max}$ ), charge transfer of TM dopants calculated by Bader charge analysis (Q) [25]

	System	$E_f$ (eV)	$Q_{c-max}$ ( $\mu F/cm^2$ )	Q(e)		System	$E_f$ (eV)	$Q_{c-max}$ ( $\mu F/cm^2$ )	Q(e)
NTM-G	NTiG	-2.52	239.84	1.665	N2TM-G	N2TiG	-4.14	163.33	1.526
	NVG	-2.44	82.87	1.324		N2VG	-3.74	133.82	1.326
	NCrG	-2.22	53.51	1.201		N2CrG	-3.53	130.15	1.195
	NMnG	-2.34	77.22	1.225		N2MnG	-3.67	87.88	1.233
	NCoG	-2.61	105.06	0.862		N2CoG	-3.98	132.69	0.792
	NNiG	-2.67	156.17	0.646		N2NiG	-4.05	177.75	0.647

To better understand the effect of doping number of N atoms on the structural stability, the local density of states (LDOS) was analysed for Co, and other transition metals follow the same properties as Ni [28]. There is obvious hybridization between the doped TM atom and N atoms; the more of the number of doped N atoms, the stronger the hybridization. As a result, the DOS gradually shifts to the lower energy levels as the doping number of N atoms increases. This DOS behaviour indicates that the more numbers of doped N atoms means a high stability for TMN<sub>x</sub>-G, which is consistent with the analysis results of  $E_f$ . [28]

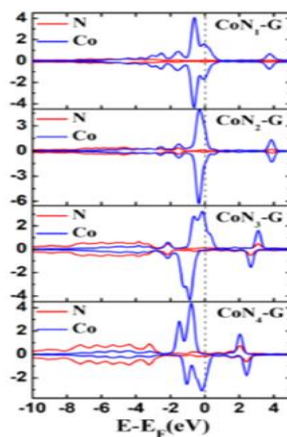


Fig.2. Local density of states of the CoN<sub>x</sub>-G systems [28]

In addition, the electronic charge distribution of doped TM atoms calculated by Bader charge analysis was analysed, and the results are listed in Table 2 above. It shows that the

more the doping number of N atoms, the more charges the TM atoms transfer. This can be explained that the electronegativity of N atom (3.04) is greater than that of C atom (2.55) [28]. For the same element, a remarkable charge transfer means a stronger bonding between the charge transfer and the surrounding atoms, thus a more stable structure is formed. As a result, increasing the doping number of N atoms is obviously an effective way to improve the stability of graphene-based materials, which is consistent with the analytical results of  $E_f$  and DOS.

## 5.2 Quantum capacitance

Upon co-doping, the Quantum Capacitance ( $Q_c$ ) and charge transfer of TM dopants calculated by Bader charge analysis (Q) of TMN<sub>x</sub>-G increased dramatically in comparison with pristine graphene. The co-doping of TMN<sub>x</sub> has resulted in an increase in the Q from 18.29  $\mu\text{C}/\text{cm}^2$  to 111.06  $\mu\text{C}/\text{cm}^2$ , and a maximum in  $Q_c$  from 40.26  $\mu\text{F}/\text{cm}^2$  to 180.50  $\mu\text{F}/\text{cm}^2$ . When TMN<sub>x</sub> is co-doped, localised states are introduced around the Fermi level. This increases the number of accessible states around the Fermi level, which can improve the  $Q_c$  of materials based on graphene. Similar to the DOS of CoN<sub>x</sub>-G displayed in Figure 2, CoN<sub>2</sub>-G exhibits a unique DOS peak at around -0.3 eV. It is discovered that other transition metals are also contributing significantly to the improvement of quantum capacitance in other doped systems [25]. Table 2 also summarises the charge transfer of TM-dopants in order to comprehend the mechanism of co-doping improvement of quantum capacitance. Positive Q values show that the TM-dopants contributed electrons to the system. In terms of enhancing quantum capacitance among the four varieties of co-doping graphene substrates, the Ti-doped structures provide the best results. In the meantime, Ti-dopant has a higher Q than the other five varieties of TM-dopant. The Q of the N-dopant in NTiG is 2.274e, whereas the Q of the Ti-dopant is 1.665e. In the N<sub>2</sub>TiG system, the two N-dopants accept the charge of 2.345e and 2.265e, respectively, whereas the Ti-dopant loses 1.526e of charge [25]. The results of Q show that the co-doping of N and TM-dopant enhances the substrate's charge transfer, which helps to increase quantum capacitance.

## 6. SUMMARY

The key to efficient supercapacitors is the development of electrode materials with high conductivity and large specific surface area which often facilitate fast charge transfer and ion diffusion that leads to improved rate-capacitance performance. In this review, analysing density functional theory calculations, the effects of TMN<sub>x</sub> co-doping on the stability, density of states, quantum capacitance and surface charge density of graphene are systematically investigated. The formation energies, average bond lengths between the transition metals and the nearby atoms, and are in the order of CrN-G > MnN-G > VN-G > TiN-G > CoN-G > NiN-G and TiN<sub>x</sub>-G > CrN-G > VN-G > MnN-G > CoN-G > NiN-G respectively for graphene with different dopant transition metal, while the order are: TMN<sub>1</sub>-G > TMN<sub>2</sub>-G > TMN<sub>3</sub>-G > TMN<sub>4</sub>-G for graphene with various N atoms. By the

co-doping of TMN<sub>x</sub>, the quantum capacitance and surface storage charges of TMN<sub>x</sub>-G are significantly increased. The DOS of TMN<sub>x</sub>-G determines its quantum capacitance, and the co-doping of TMN<sub>x</sub> introduces localised states around the Fermi level, which leads to the increase of available states around the Fermi level. The doping of TM plays an important role in the enhancement of quantum capacitance because the introduced impurity states around the Fermi level are mainly contributed by doped transition metals. In addition, the quantum capacitance and surface storage charge are enhanced monotonically with the increase of doping concentration, but the stability decreases. The present study indicates that TMN<sub>x</sub> co-doped graphene has an excellent application potential in the electrode materials of supercapacitors. The research results can provide design strategies and theoretical basis for designing graphene-based supercapacitors with high capacitance.

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