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Textural Properties of Electrospun Magnesium Oxide (MgO) Nanofibers

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ABSTRACT

The optimised morphology of electrospun magnesium oxide (MgO) nanofibers (NFs) can be achieved according to the parameters set during the fabrication process. However, not all materials can be synthesized together as it depends on the application needs. This research aims to study the factors that influence the morphology of the MgO NFs due to material preparations and electrospinning parameters. The research is based on data obtained from previous research papers that were analysed using MATLAB R2019a software to evaluate the effect on MgO nanofibers synthesized with different materials. The study was focused on textural properties of nanofibers such as pore-volume and fiber diameter. Based on the analysis obtained, researchers need to consider using polymer material during the electrospinning fabrication. Each type of the polymer used will produce different values of the MgO NFs diameter after calcination process. Poly-propylene glycol polymer showed a very significant difference in the fiber diameter size in the range of 60 to 76 nm as compared to polylactic acid produced big size of fiber diameter in between 3300 to 6900 nm. The analysis showed that the use of nitrogen as a carrier gas in the fabrication of electrospun MgO NFs may produce a good quality of the nanofibers.

Keywords: *Electrospun, magnesium oxide, MATLAB, pore volume and fiber diameter.*

1. INTRODUCTION

Nanofiber materials that can be used in the fabrication of electrospinning techniques includes metal, metal oxide, carbon, inorganic, composites and polymers where their diameters may range from tens of nano meters to several micro meters [1]. Electrospinning which produces a patent of continuous fibers was first developed in 1934 [2]. Since then, the technique has gained much interest from other researchers to study and explore its application. Electrospinning is a versatile technique as it can be used to fabricate one-dimensional (1D) nanomaterials. The nanofibers prepared by electrospinning method is known to have larger specific surface areas, higher aspect ratio, and the interconnectivity of pore is better compared to other techniques. This makes the technique more promising for energy transformation and storage applications. The morphologies and structures obtained through this method include core-shell, hollow [3], yarn, and porous [4] structures. These structures could be obtained by modifying the electrospinning properties such as applied voltage, feed rate flow, collector type, tip-to-collector distance, nozzle design, and calcination treatment.

It has been researched that magnesium oxide (MgO) and aluminium oxide (Al_2O_3) that act as catalysts have the same effect on carbon precipitation, but MgO is much easier to be eliminated

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compared to Al_2O_3 [5,6]. MgO is a white hygroscopic solid mineral that consists of magnesium ions and oxygen ions bonded together. In general, MgO is produced by going through the calcination process of magnesium hydroxide ($Mg(OH)_2$) or magnesium carbonate ($MgCO_3$) [7,8]. Nanoscale MgO is suitable in applications that involved thermodynamic, optical, electronic, and special chemical properties [9]. Magnesium oxide is one of the non-magnetic oxides that has been popular for the past few decades due to the existence of several phenomena in nanostructures [10,11]. The phenomena that have been observed in its nanostructures include cataluminscence, spin-dependent reflectivity, high dielectric constant, magnetism, laser transition, reduced optical bandgap, electroluminescence, and luminescence. The nanofibers of MgO play a significant role in numerous fields such as medical, electrical, and magnetic applications, and environmental [9]. MgO is an important material as it is suitable in the application that involved catalysis, water treatment, refractory material, photocatalysis, and carbon dioxide (CO_2) separation adsorbent from the flue gases [10].

Nanofibers exhibit numerous properties that can be used in several sectors such as optics, electricity, mechanics, thermotics, magnetics, and chemistry. This is because nanofibers are known for their surface effect, small-size effect, interface effect, and quantum size effect. MgO nanofibers between the range of 0.1 to 100 nm play an important role in prospective filler as they can sustain high voltage electric [12,13]. Most of electrospinning materials are based on magnesium (Mg) compounds, which includes fabrication of MgO nanofibers (NFs) and magnesium nanoparticles (NPs) obtained by calcination process [9]. Normally nanofibers produced by electrospinning have different structures such as orientation and hierarchical structures. The arrangement of nanofibers is either random or aligned structures. The well-aligned structures of nanofibers that have been fabricated are often applied for energy devices application [3]. The alignment of nanofibers can be enhanced by using a specially designed collector. The collector can be either a rotating drum, metal frame, or two conductive substrates that are separated by a gap.

The characteristics of electrospinning of nanofibers depend on several specifications. These parameters are divided into three categories namely the solution parameters, process parameters, and environmental conditions. The solution parameters involved are the concentration and viscosity of the solution, solution surface tension, and its conductivity. Solution concentration is important to determine the diameter of fibers. Nanofibers that have a smaller diameter size can be obtained by reducing the concentration of the solution used [2]. However, beaded fibers will be formed when the concentration decreased to the entanglement concentration [2]. The fabrication of nanofibers however faced great challenged to obtain well-defined morphology with high yield. This is due to the researchers need to consider each detail of the materials and parameters that need to be set. Physical and environmental affect the nanofiber morphology. The physical condition that needs to be taken care of was the electrospinning parameters that include the speed of the collector, flow rate, and voltage applied to the needle [2]. The environmental parameters that might affect the formation of nanofibers are the ambient conditions, humidity, and condensation [2].

The research conducted by Islamic Azad University in 2017 involved the fabrication of MgO and polypropylene glycol (PPG) to form hybrid nanofibers for the studies on the removal of heavy metal ions [5]. MgO is known to have great ability in metal adsorption due to its properties that can precipitate heavy metal ions. The adsorption capacity of heavy metals obtained by mesoporous MgO nanofiber was 378.58 mg/g for plumbum (Pb), 311.47 mg/g for

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cadmium (Cd), and 270.11 mg/g for copper (Cu) [8]. However, the adsorption capacity increased when MgO nanofibers had been doped with PPG. The mesoporous MgO/PPG adsorption capacity became 2500.48 mg/g for Pb, 2407.74 mg/g for Cd and 2415.74 mg/g for Cu [8].

The degradation activity between the fabrication and conventional MgO nanospheres were compared in the research conducted by Mantilaka et al. in 2018 on the fabrication of nanofibers by using the electrospun method using polyvinyl alcohol (PVA) with magnesium precursor-based system [14]. It is reported that with the presence of photocatalytic MgO nanofibers, complete degradation of the reactive dye can be obtained under the influence of ultraviolet (UV) irradiation for 100 minutes [14].

In this study, the detailed process on the morphology of MgO nanofibers was obtained from the selected research papers [2,10-15]. The collected data were then analysed using the MATLAB R2019a software by plotting the graph according to the parameters used and characteristics of the formed nanofibers. It is important to consider the type of additive substances in the fabrication of MgO nanofibers according to its application. Based on the collected data, the textural properties of electrospun MgO NFs were analysed and studied.

2. METHODOLOGY

The study works were conducted to determine the textural properties of MgO nanofibers that have been fabricated through the electrospinning method. The study focused on the electrospinning method to fabricate the nanofibers where it used a high voltage power supply, grounded collector, and positively charged capillary that contains polymer fluid to produce fibers up to diameter less than 100 nm. All the analysis for pore-volume and fiber diameter were done in order to obtain the morphologies of nanofibers. The research started off with the process of collecting data from the previous research papers [2,10-15] on the formation of the MgO nanofibers via the electrospinning process till the graph plotting process is completed using the MATLAB R2019a software.

The collected data were divided into other parameters so that the textual properties can be easily observed. First, the data were collected for organic solvents; ethoxide (ME), magnesium oxalate dihydrate (MO), magnesium lactate dihydrate (ML), magnesium citrate (MC), magnesium gluconate hydrate (MG), and magnesium acetate (MA). Then the collected data were focused on the MA with the different concentration of MA. Finally, the data were plotted accordingly and each of the data was analysed according to the MgO NFs morphologies and applications.

3. RESULTS AND DISCUSSION

The analysis and discussion of the data obtained from the previous research papers [2,10-15] were explained in this section. The obtained data show a variation of results in the formation of MgO nanofibers. This phenomenon happened as the effects of the materials or polymers that were mixed into the MgO solvents. The categories studied are pore-volume and fiber diameter.

3.1 Pore Volume

The collected pore volume data from literature were analysed utilising the Barrett−Joyner−Halenda (BJH) methods. BJH method is used to determine the pore area and

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specific pore volume by using the adsorption and desorption technique. Some organic solvents that were used in the production of MgO nanofibers are MO, ML, MC, MA, MG and ME. The pore volume distribution of different MgO sorbents namely MO, ML, MC, MA, MG and ME are particularly between the range of 0.17 to 0.42 cm^3/g as in Figure 1 [10]. MO sorbents achieved greater volume of up to 0.42 cm^3/g, following by ME sorbents with pore volume of 0.3189 cm^3/g [10]. Each of the organic solvents has its own viscosity. Although viscosity has an important role in the formation of fibers, the initiation of electrospinning depends on another important factor which is the surface tension. The charged solution has to overcome the surface tension in order to form the polymer jet. If the solution has low viscosity, a high tendency for the solvent molecules to form a spherical shape (beads) will be observed due to the action of surface tension which also will affect the values of MgO NFs pore volume [11]. This is the main reason why each of the organic solvent has resulted in a different pore volume.

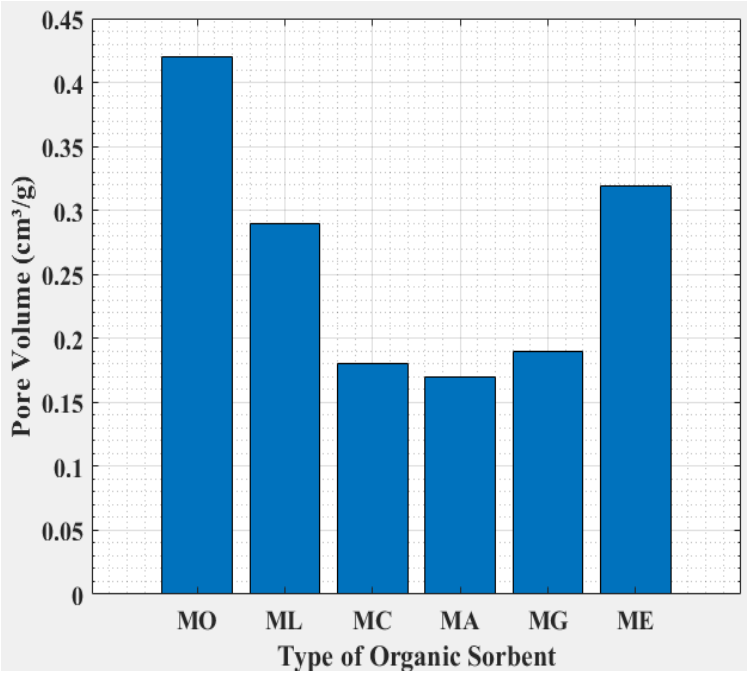

Figure 1: Pore volume of MgO nanofibers obtained with different organic solvents.

Figure 2 shows a plot of pore volume data for the MA sorbents with different concentrations. The concentration of MA solution is made by varying the percentage composition of Mg by 1% from MA-1 to MA-6. The main reason for focussing more on MA sorbents because it is more often used in electrospinning process of MgO NFs [12]. The pore volume of the MA sample with different concentration increased from 0.22 for MA-1 to 0.27 for MA-4. MA-6 shows the highest pore volume of 0.34 cm^3/g. This value indicates that the performed structure of MA-6 is similar to the mesoporous MgO structures (0.24 to 0.37 cm^3/g). However, there was a small decrease in the volume for MA-3 and MA-5 due to the process during the conducted experiment. Each sample was heated at a different temperature from 349.85 ℃ to 599.85 ℃ [12].

Table 1 shows the detail values of the pore volume and average pore diameter for each of the adsorbent N-500, A-500, C-500 and C-400 during the calcination process condition [13]. From the data it can be seen the MgO-C pore volume and average diameter had decreased as the

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heating temperature was decreased from 500°C to 400°C. It should be noted that the MgO-N has the highest value of pore volume (0.68 cm^3/g) and smallest average diameter (6.16 nm). The experiment was designed to study the effects of calcination atmosphere and temperature on the MgO adsorbent.

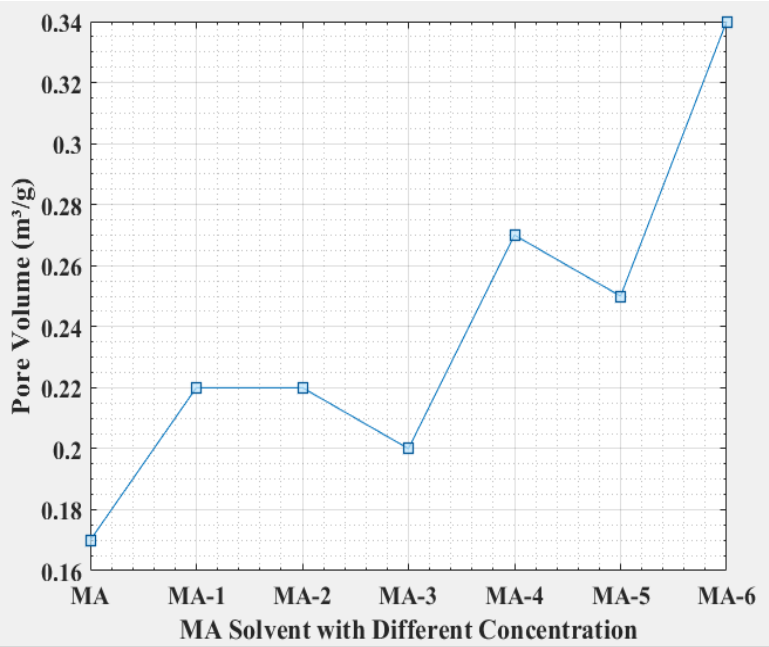

Figure 2: Pore volume with different MA solvent concentrations.

Table 1: Pore volume and average pore diameter for different type of adsorbents [13].

Adsorbents	Calcination Condition	Pore Volume (cm^3/g)	Average Pore Diameter (nm)
N-500	Flowing N_2 (100 ml/min) at 500 °C, 10 °C/min for 60 min	0.68	6.16
A-500	Static air at 500 °C, 10 °C/min for 60 min	0.42	24.14
C-500	Flowing CO_2 (100 ml/min) at 500 °C, 10 °C/min for 60 min	0.53	20.40
C-400	Flowing CO_2 (100 ml/min) at 400 °C, 10 °C/min for 60 min	0.17	21.15

Figure 3 shows additional data for MgO-C-450, MgO-C-550 and MgO-C-600 as compared to the data in Table 1. The MgO-N-500, MgO-C-500 and MgO-C-450 adsorbents show significantly larger pore volumes compared to others as seen in Figure 3. This happened since the flowing of N_2 and CO_2 removed the discharged water vapor quickly. It also reduced the grain impurities and blockage of the pore structure. The low pore volume of MgO-C-400 was caused by the incomplete decomposition of the magnesium oxalate dihydrate. Pore expansion occurred due to gas expansion which resulted from the increased surface area and tighten porous structure. However, the textural parameters that can be seen in Figure 3, decreased sharply when the precursor was under high calcination temperature at 550 °C and 600 °C. This was due to the grain sintering that affects the blockage of the pore structure [13].

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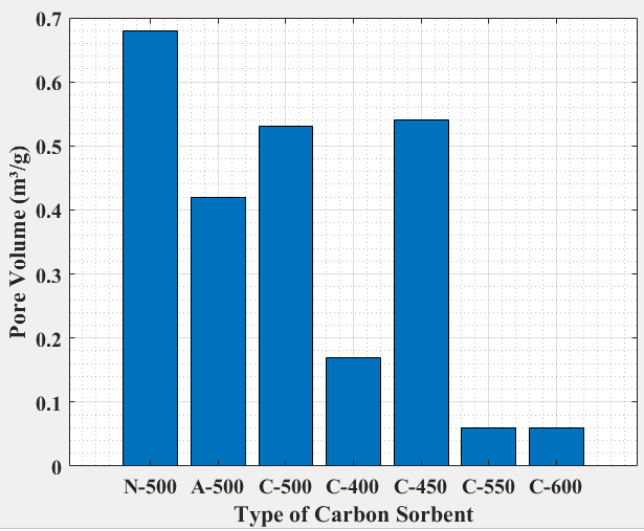

Figure 3: Pore volume at different calcination atmosphere and temperature.

3.2 Fiber Diameter

The main electrospinning parameters for obtaining the diameter of the fibers according to the various type of polymers are shown in Table 2. The information in this table is important for researchers to optimize each of the parameters to obtain high-efficiency nanofibers and high capability to remove the environmental contaminants.

Table 2: Parameters applied in the experiment [2].

Polymer	NPs	Electrospinning Parameters			Post-Electro-spinning Process	Fibers Diameter (nm)
		Voltage (kV)	Distance (cm)	Flow Rate (mL/h)		
Poly-propylene glycol (PPG)	MgO	21	15	0.5	Calcination	60 - 76
Polyvinyl alcohol (PVA)	MgO	26 - 28	10	0.5	Calcination	35 - 200
Alginate-PVA	MgO	26 - 28	10	0.08 – 0.01	Direct	60 - 250
Polyvinyl-pyrrolidone (PVP)	MgO	-	6	-	Calcination	180 - 260
Poly-acrylo-nitrile (PAN)	MgO	12	20	1	Calcination	316.6 ± 41.72
Poly-urethane (PU)	MgO	10	20	0.2	Direct	From 522 ± 159.10 to 622 ± 174.75
Cellulose acetate-Poly-ethylene oxide (PEO-CA)	MgO	16 - 18	15	0.04 – 0.08	Direct	1350 ± 390
Polylactic acid (PLA)	Bio glass with MgO	16	20	0.15	Direct	3300 - 6900
Polycapro-lactone (PCL)	MgO	19	-	1.9	Direct	200 - 600

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Polymer solution and the molecular weight of the polymers are found to be the most significant in the formation of the fibers. During the electrospinning process, a sufficient molecular weight of the polymer is required to increase the entanglement between polymer chains which are crucial for the continuity of the jet formed during the electrospinning process. Polymer-based MgO NFs are important materials for research and industrial areas. Polymer MgO NFs with diameters less than 1 µm has unique properties including large specific surface area per unit mass, which facilitated adding functionalities to surface for specific applications. The fiber diameter with polymer polylactic acid (PLA) loaded onto bio-glass with MgO has the largest diameter which was between the range of 3300 to 6900 nm compared to the others. Polymer PPG however showed a very significant difference in the fiber diameter size in the range of 60 to 76 nm as it was suitable in the removal process of heavy metals [8].

A Group experimented with 15 samples of polyacrylonitrile (PAN) with different electrospinning parameters to predict the optimum condition of the respective method [15]. They stated that PAN is a versatile polymer as it has good physical characteristics, is affordable, and the electrospinning process is easier to handle [15]. The average fiber diameter can be concluded in Table 3 [15].

Table 3: Textural properties of the different PAN samples [15].

No. of Samples	Electrospinning Parameters			Average Fibers Diameter (nm)
	Concentration (wt%)	Applied Voltage (kV)	Tip-to-Collector Distance (cm)	
1	16	20	10	263.33
2	16	10	15	313.58
3	8	20	15	69.05
4	8	20	10	46.44
5	9.6	15	12.5	27.78
6	14.4	15	12.5	113.39
7	12	12	12.5	105.51
8	12	18	12.5	45.36
9	12	15	11	43
10	12	15	14	98.5
11	12	15	12.5	54.8
12	12	15	12.5	68.1
13	12	15	12.5	84.36
14	12	15	12.5	72.4
15	12	15	12.5	52.22

The maximum average fiber diameter was 313.58 nm, obtained from the experiment on sample number 2. Sample number 2 has a concentration of 16 wt% with electrospinning parameters of 10 kV of applied voltage and 15 cm distance. Experiment on sample number 5 obtained minimum average fiber diameter at 27.78 nm with a concentration of 9.6 wt% and electrospun parameters of applied voltage at 15 kV with (or within) a 12.5 cm distance. Even though with lower concentrations, this sample obtained smaller diameters of fiber, the fiber might not be of high quality. The composite nanofibers can be increased by changing the PAN polymer into polyvinylpyrrolidone (PVP) polymer. The studies showed that the average fiber diameter at higher concentrations was larger than at lower concentrations. This happened as more polymer chains can entangle but the mobility of chain is reduced causing strong jet extension and higher disturbance during the electrospun process [15].

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Table 4 shows the fiber diameter obtained when polymer polycaprolactone (PCL) was doped with other polymers. The addition of natural polymer keratin to PCL when doped with MgO increased the ultimate tensile strength higher than pure PCL by 3.3% [16]. This is due to the nature of keratin that can reappear in the integrin-binding domains. PCL-Keratin is known to have the potential in vascular tissue engineering [17]. An overview of the electrospun parameters and their mechanical properties are summarized in Table 4 [17].

Table 4: Textural properties of the different PCL polymers [17].

Polymer	NPs	Electrospinning Parameters			Post-Electro-spinning Process	Fibers Diameter (nm)
		Voltage (kV)	Distance (cm)	Flow Rate (mL/h)		
Polycaprolactone (PCL)	MgO	19	-	1.9	Direct	200 - 600
PCL-Keratin	MgO	11	10	1-2	Direct	200 - 2200
PCL-CS	MgO	25 - 27	7	2.5	Direct	60-250
PCL-CC	SiO_2-Na_2O-K_2O-MgO-CaO-P_2O_5	17	-	1	Deposition of polymer fibers on bioactive glass fibers	2000 ± 2300 glass 750 ± 540 PCL
PCL	LDHs	20	20	-	Direct	350 ± 50 PCL-LDH organic-ally modified

PCL-CS/MgO solution was prepared by mixing the chitosan (CS) solution drop-wise into the solution of PCL and MgO. The PCL-CS/MgO composites have the advantage of integrating good biological properties of chitosan and good mechanical properties of PCL. This is because both polymers do not need chemical crosslinking to maintain their structures and preferable mechanical properties. Chitosan also produced a small fibrous network formation than other nanofibers obtained from other polymers [18].

The PCL-CC was a mixture of silicon dioxide (SiO_2), sodium oxide (Na_2O), potassium oxide (K_2O), MgO, calcium oxide (CaO), and phosphorus pentoxide (P_2O_5) which are bioactive glass microfibers. This type of bio-composite was suitable for nerve guide application [19]. The advantage of this bio-composite was that it is permeable to water vapor, which permitted the exchange of nerve growth factors [19]. The PCL-LDHs means that the layer of PCL was simulated with layered double hydroxides (LDHs). The structure of LDHs was made up of magnesium atoms that were surrounded by a hydroxyl group. The average diameter of PCL-LDHs nanofibers was 600 ± 50 nm.

4. CONCLUSION

As a conclusion, the research was successfully done by tabulating the collected data from previous research papers on MgO NFs using electrospinning technique and analysing all data using MATLAB R2019a software. The graphs were generated according to different textural properties (pore volume and fiber diameter) that are affected from the electrospinning fabrication process. The analysis results showed that the morphology of MgO NFs was affected by the concentration of sorbent and the calcination conditions (atmosphere and temperature)

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used during the electrospinning process. Based on the analysis obtained, researchers need to consider using polymer material during electrospinning fabrication. Each type of the polymer used will produce different values of the MgO NFs diameter after the calcination process. PPG polymer showed a very significant difference in the fiber diameter size in the range of 60 to 76 nm as compared to PLA produced big size of fiber diameter in between 3300-6900 nm. The size of fiber diameter produced is important since different applications will be required different size of fiber diameter. For example, the fiber diameter for PCL-Keratin is up to 2200 nm and it is suitable for vascular tissue engineering application. While for PCL-CS nanofiber, the fiber diameter is 250 nm which is suitable for nerve guide application. This study also suggested that for intermediate temperature CO_2 capture applications, it is suitable to use carbon instead of organic solvent. The organic solvent is thus, suitable for the sustainability of the environment as the cost for fabrication can be reduced and is environmental friendly.

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