# Industrial Oriented Approach on Fullerene Preparation Methods

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# Industrial oriented approach on fullerene preparation methods

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ABSTRACT

Fullerene, a new allotrope of carbon with a spherical-shaped molecular structure, draws substantial attention of nano-scientists nowadays because of having numerous applications in different industrial fields such as materials science, building nano-sensors, modified composites, molecular drug containers, etc. This is the reason why having knowledge about fullerene is essential for any scientist working in any related field. Although fullerene is highly applicable, the high costs of its production has prevented its widespread use. There are numerous fullerene synthesis methods introduced by scientists, any of which has its own benefits, disadvantages, production costs, and parameters to be improved. In this review, fullerene preparation methods set up by various scientists were introduced and production process of each method was explained. Afterwards, each method and its important parameters, along with its direct effect on production yield and ways of improvement for each parameter, were discussed. Finally, all the methods were compared with one another, followed by selecting the methods which appeared to be economically justifiable for use in mass production.

## 1. Introduction

In 1985, a new and stable allotrope of carbon consisting of 60 carbon atoms with a spherical-shaped molecular structure named fullerene was discovered by Kroto and co-workers after vaporization of a carbon source [1]. Since then, many researchers have discussed various applications of fullerene in different industrial fields such as materials science [2–4], building nano-sensors [5–7], modified composites [8–10], molecular drug containers [11–14], organic solar cells [15–17], water filters [18,19], electro-catalysts [20], battery electrodes [21], etc. These scientific works raised the importance of fullerene production, which resulted in appearance of numerous fullerene synthesis methods [22–25].

Although there are numerous synthesis methods introduced, fullerene production prices are still high, but this is not the main issue [26]. There are many issues with fullerene production methods that keep production prices high enough to prevent widespread use of fullerene [27]. The main issues are high costs of fullerene production processes applied in each method, very low yields of production, and high costs of fulerene seperation methods employed after production. The main reason for high prices of production is very low yields and high costs of separation methods, and the reason for low yields in nearly all the methods is that there is no absolute scientific explanation for fullerene formation in each method, nor is there chemical explanation for fullerene formation either [28–31]. With all these in mind, scientists all over the world have discussed numerous synthesis methods, because fullerene has incredible applications in nearly all of the industries, eventhough nearly all of the methods are costly. So far, no method has contributed to producing fullerene at low prices, so scientists have attempted to offer and discuss the improvement of parameters in each fullerene production method rather than seeking new synthesis methods [32,33]. Generally, science of fullerenes and carbon nanotubes [34–36] drives chemists and materials scientists to the field of fullerenes, and presents the unique properties and applications.

In this review study, first we explained all of the fullerene synthesis methods considered appropriate to become industrialized such as arc-discharge, combustion, laser ablation, and microwave reactors. Then we discussed important parameters having direct effects on fullerene

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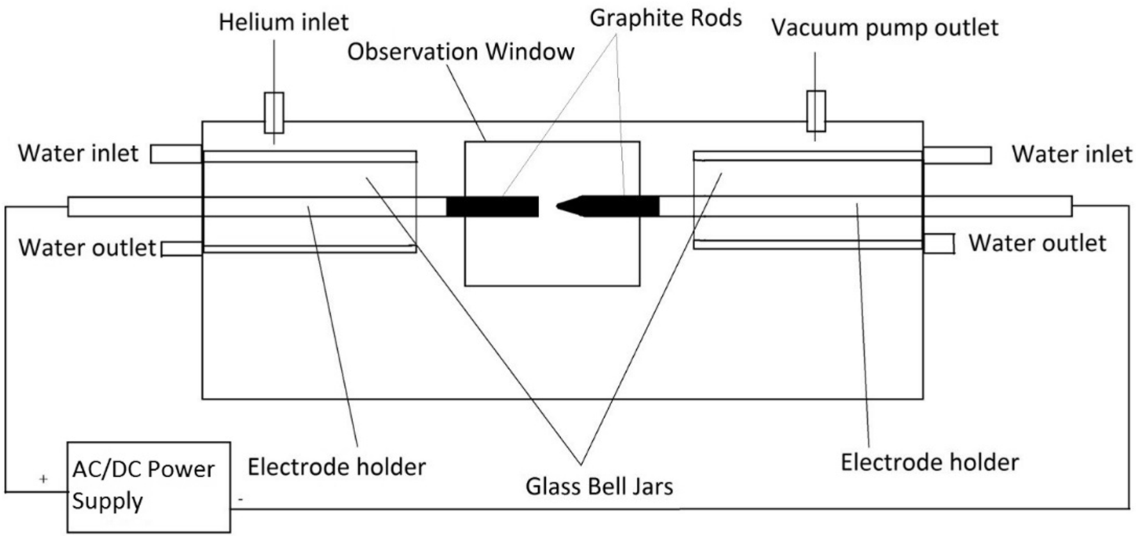


**Fig. 1.** Arc-discharge reactor apparatus.

production process in these methods. Also, we elaborated how important parameters in each synthesis method can be improved. Finally, we compared the results and introduced the method, among all of the discussed methods, with high potential to be an industrially justifiable method of fullerene production in the future.

## 2. Synthesis of fullerene by arc-discharge

### *2.1. Synthesis process initials*

Synthesis of fullerene using arc-discharge is based on vaporizing a carbon source by direct or alternative electrical current in an envinronment which is neturalized by a low pressure innert gas such as helium and argon. This is an effort to reach very high temperatures without causing damage to the reactor used in the process, and to prevent carbon source from oxidation. The main parameters which have direct effects on production process in this section include very high temperature, low gas pressure, electrodes gap, electrodes thickness, and low gas velocity [37–45].

### *2.2. Arc-discharge reactor apparatus*

There are many different arc-discharge apparatus units used to synthesize fullerene, the most common unit of which is the tube-shaped. The tube-shaped reactor consists of a tubular space made of stainless steel to ensure rigidity and strength while confronting external air pressure, two electrode holders, two water-cooled glass bell jars placed around the electrodes, two water inlets and two water outlets for bell jars which are accessible from outside the reactor, an innert gas inlet, a vacuum pump outlet, a plasma observation window, and an AC/DC power supply connected to each electrode end from outside the reactor [46]. The reactor apparatus is shown in Fig. 1.

### *2.3. Carbon source*

Carbon source used in this method is mainly graphite, but carbon purified coal can also be used in the process. The reason why mainly graphite is used in this process is that graphite has quite high electrical resistance enabling us to initiate arc-discharge between two graphite rods. This causes the high temperature in which bright plasma can be obtained.

### *2.4. Neutralized environment*

In order to neutralize the internal space of reactor, low pressure inert gas such as helium or argon is pumped inside the reactor space after removing the air from the reactor under vacuum. Using a low pressure innert gas has three benefits for the production process, including creating a neutralized environment in which carbon source will only vaporize, reaching low plasma speed due to low gas pressure inside the reactor which will improve production yield, and enabling the graphite rods to reach to high temperatures without melting the reactor walls [41,47].

### *2.5. Production process explanation*

The production process starts with filling up the generator with Helium or Argon, and adjusting the gas pressure to reach 100 Torr, then pushing the graphite rods together leaving no space between them. Then the current is set up to 70 A and the voltage is set up to 20 V. After that, resistive heating occurs when the rods are touching. Then the rods are pulled apart to make a tiny gap ($\approx$ 1 mm) between them and the pulling process is kept until the Helium or Argon plasma starts glowing steadily (there shuoldnt be a change of color of plasma). After obtaining bright colored plasma, graphite rods start to vaporize until the space between the tips of the rods is big enough to stop the arching process. At this point, the temperature of each rod reaches 4000 °K so the rods should be cooled down as the process proceeds using the water cooled glass bell jars around each rod; if the current is direct, one of the rods will vaporize and only the vaporizing rod should be cooled down but if the current is alternative, then both of the rods will vaporize and therefore both of them should be cooled down. Perhaps by measuring graphite vaporization speed and designing a system to move the electrodes at same speed steadily, the consumption rate of graphite rods will be more economically justifiable. After the vaporization of graphite rods, if the current is direct, it is necessary to remove the light-colored slag from the electrode which has not vaporized in the process. When the process is finished, the reactor should be filled with atmospheric pressure of Helium or Argon and allowed to cool down. When the reactor is cooled down, the soot produced in the process can be collected using the vacuum pump outlet or by opening the chamber and removing the collector shims and scraping the soot from shims.

**Table 1**
Results of soot purification by Soxhlet method [53].

| Number of run | Solvent | Temperature (°C) | Fullerenes extracted (g) | Fullerenes extracted (%) | Efficiency (%) |
|---|---|---|---|---|---|
| 1 | Carbon disulfide | 57 | 0.34 | 6.8 | 97.14 |
| 2 | NMP | 215 | 0.08 | 1.6 | 22.86 |
| 3 | Chlorobenzene | 142 | 0.352 | 7.04 | 100.0 |
| 4 | 1,2-Dichlorobenzene | 190 | 0.357 | 7.14 | 100.0 |
| 5 | 1,2,4-Trichlorobenzene | 225 | 0.357 | 7.14 | 102.0 |
| 6 | 1-Methylnaphthalene | 255 | 0.357 | 7.14 | 100.0 |
| 7 | Dimethylnaphthalenes | 280 | 0.357 | 7.14 | 100.0 |
| 8 | 1-Phenylnaphthalene | 335 | 0.357 | 7.14 | 100.0 |
| 9 | 1-Chloronaphthalene | 275 | 0.357 | 7.14 | 100.0 |
| 10 | Toluene | 120 | 0.16 | 3.2 | 45.71 |

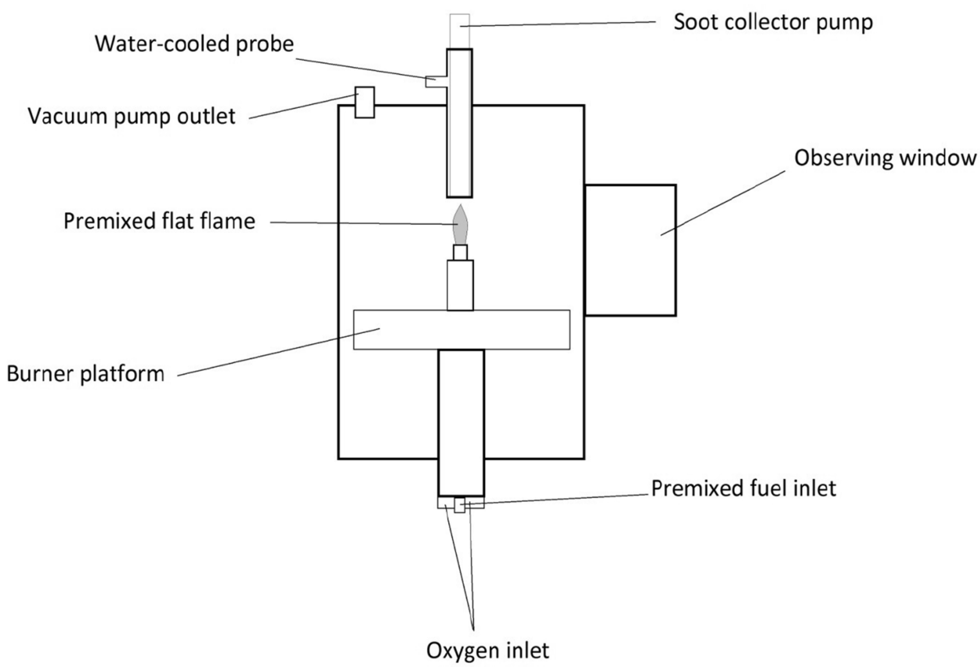


**Fig. 2.** Combustion fullerene generator system.

### *2.6. Soot purification process*

The purification process can be carried out either by traditional soot washing using hyrocarbons such as hexane, or applying Soxhlet method solely [48], or assisted one [49], using light hydrocarbons such as benzene or toluene for small fullerenes (C60), or heavy hydrocarbons such as hexane or heptane for large fullerenes (Cn , n > 60). The results of scientific works show that purification process carried out by Soxhlet method appears to reach higher yields of fullerene preparation than the traditional purification process, which are showed in Table 1 [50–52].

## 3. Synthesis of fullerene by combustion

### *3.1. Synthesis process initials*

Synthesis of fullerene by combustion is based on igniting a mixture of oxygen, a noble gas such as argon, and a hydrocarbon as a fuel inside a generator and collecting the soot including fullerenes and other nanostructures such as different kinds carbon nanotubes, nano-onions and etc produced in fuel combustion process and applying a purification process on the soot. Contrary to what was described before, the generator is not filled with a noble gas prior to fullerene production process. Instead, the air is vacuumed out of generator space in order to create a neutralized environment, and avoid causing damage to reactor space due to very high temperatures. The vital parameters that have direct effect on fullerene production in this method and need to be improved are very high temperature, low gas pressure inside the generator, burner platform height, and speed of soot pumping process [54].

### *3.2. Fullerene genrator system*

The generator used in this production method consists of a tube-shaped space made of stainless steel to ensure endurance while confronting air pressure on the outside surface of the generator. Also, the dimensions of this tubular space are different than arc-discharge generator system because there should be enough distance between the burner and the tube walls to avoid melting of the walls while reaching very high temperatures in the middle of the soot production process. The generator consists of a tube-shaped space as mentioned above, a soot collector pump, a water-cooled probe around the soot collector pump, a vacuum pump outlet, an observing window, a burner platform, a premixed fuel inlet, and an oxygen inlet [55]. The genrator sytem used in this production method is shown in Fig. 2.

### *3.3. Carbon source*

Carbon sources used to produce soot containing fullerenes are mainly light or straight-chained hydrocarbons to ensure runing incompelte combustion process and reaching high temperatures, but in most combustion reactors premixed benzene/oxygen fuel is used to produce

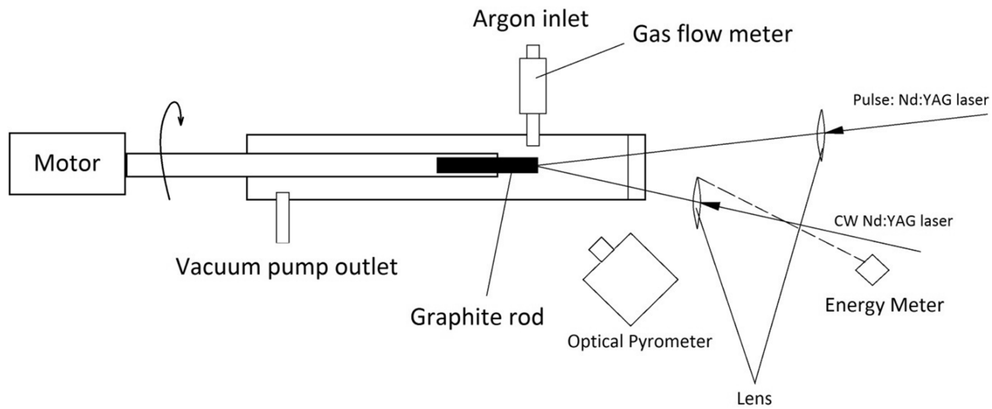


**Fig. 3.** Laser fullerene generator system (graphite as carbon source).

soot [55–57].

### 3.4. Neutralized environment

Combustion reactor has critical temperature-related issues. One of the main issues is that in order to reach fullerene production point, the premixed benzene/oxygen/argon flame tip should reach very high temperatures. However, reaching very high temperatures will cause serious damages to reactor space like melting down the reactor walls; therefore, the neutralized environment must have lowest possible thermal conductivity to avoid damages. That is the reason why the reactor space is not filled with a low pressure noble gas such as helium or argon. Instead, all of the air is pumped out to ensure the lowest thermal conductivity of production environment using a vacuum pump [55,58,59].

### 3.5. Production process explanation

The production process starts with vacuuming all the air out of generator space. At this point, benzene/oxygen/argon premixed fuel is injected into the reactor space. Next, the burner platform ignites the injected fuel mixture and fuel starts burning. Then, the height of burner platform is adjusted in a way that the tip of the premixed flat flame reaches soot collector pump outlet with a very small distance and the soot produced from fuel combustion is simultaneously collected using water-cooled soot collector pump outlet which is located on the top of the flame tip. After producing certain amount of soot, the generator shuts down in order to cool down before starting the production process again and avoid the damage caused to reactor space after reaching very high temperatures [54,60–65].

### 3.6. Soot purification process

Just as arc-discharge method, soot purification process can be carried out either by traditional methods or Soxhlet method, but there are green methods of fullerene purification which we will discuss in the subsequent section.

## 4. Synthesis of fullerene by laser

### 4.1. Synthesis process initials

Depending on the fullerene production process type, laser, as a heat source, is used either to vaporize or combust carbon source. There are reasons why laser is used to vaporize or combust carbon source. Vaporizing a carbon source using laser is meant for two reasons: environment-friendly (green) heat source, and reaching very high temperatures just on a point which will not cause damage to reactor space, unlike the combustion process. On the other hand, the combustion of carbon source using laser has the same heat source benefit as it was mentioned above except for the reactor space which will become more vulnerable to damage caused by heat while using laser in combustion process [61,66–68].

#### 4.1.1. Synthesis by vaporization

The first synthesis process type using laser as a heat source is vaporization of carbon source. This process is carried out by vaporizing a carbon source under an inert atmosphere just the same as it was described about arc-discharge method. There should be a neutralized environment in which carbon source, mainly graphite, is able to get vaporized without burning and turn into carbon dioxide; therefore, a low pressure noble gas is injected inside the reactor space to create such an environment, after pumping all the air out of reactor space [69–71].

#### 4.1.2. Synthesis by combustion

Second synthesis process type using laser as a heat source is fuel combustion. Unlike fuel combustion method, the reactor space in laser combustion is filled with a photo-sensitizer substance, or the photo-sensitizer substance is added to premixed fuel as an energy transfer agent. Then, the premixed fuel is injected inside the reactor and ignited by pulsed laser radiation which causes the premixed fuel to burn and produce soot, respectively. Finally, the soot is collected for purification process [67,72].

### 4.2. Fullerene generator system

#### 4.2.1. Vaporizer reactor

The fullerene generator system used in this method is made of glass. The generator consists of a tube-shaped glass as reactor space, a vacuum pump outlet, a motor used to rotate the graphite rod, an argon gas inlet, an argon gas flow meter, two focus lenses placed perpendicularly to laser beams, a laser energy meter, an optical pyrometer to measures the temperature of graphite rod, and two laser generators which produce fundamental (1064 nm), second (532 nm), and third-harmonic (355 nm) light with a repetition of 10 Hz. The fullerene generator system is shown in Fig. 3.

The motor which is attached to graphite rod holder has an important duty in the production process, which is to rotate the graphite rod. This will cause constant vaporization of graphite rod in the process which has important benefits. The benefits of constant graphite vaporization are no change of direction for laser beams, producing very high temperatures just in one point so the reactor space will not get damaged due to thermal issues, constant rate of soot production, and be able to reach higher yields [69,73]. The lenses are also placed perpendicularly to laser beams to avoid laser pulse drop, and set direction for laser beams (control beam direction).

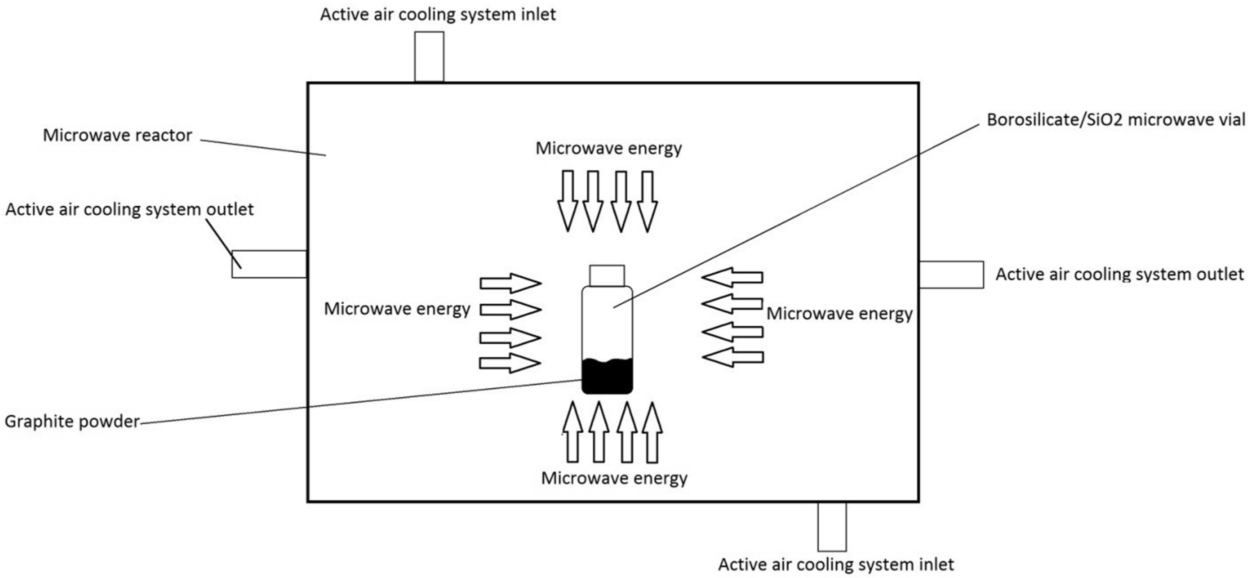


**Fig. 4.** Microwave fullerene generator system (graphite as carbon source).

*4.2.2. Combustion reactor*

The combustion reactor system is the same as the reactor used in combustion method, but with a slight difference which is about using a photo-sensitizer substance, as described in 4.1.2. Section [66].

## 4.3. Carbon source

*4.3.1. Vaporizer reactor*

Carbon source used in this method is more commonly graphite, but coal can be used in the process of soot production. The main reason why graphite is used more commonly as carbon source in soot production process is that it has high heat absorption which satisfies the wanted condition to reaching a very high temperature on a point [69,74].

*4.3.2. Combustion reactor*

There are numerous carbon sources that can be used in the combustion process [75]. All of the carbon sources are classified as light or straight-chained hydrocarbons, but mainly benzene/oxygen/photo-sensitizer substance premixed fuel is used as carbon source. The reason why benzene is used more commonly than any other light or straight-chained hydrocarbon is that it enables us to run incomplete combustion which will produce soot including fullerenes and other carbonic nano-structures [58].

## 4.4. Neutralized environment

*4.4.1. Vaporizer reactor*

As stated before, suitable neutralized environment for running vaporizer reactor is an inert atmosphere created using a low pressure noble gas such as helium or argon. Results obtained by other scientific works show that using helium to create a neutralized environment might lead to higher yields because obtaining bright-colored plasma is easier while using helium [69,74].

*4.4.2. Combustion reactor*

Unlike vaporizer reactor, suitable neutralized environment for running combustion reactor is created by pumping all the air out of reactor space. The reason for doing as such is to ensure least possible thermal conductivity of reactor space in an effort to avoid damaging the generator system while reaching very high temperatures [58,67].

## 4.5. Production process explanation

*4.5.1. Vaporizer reactor*

The production starts with vacuuming all the contents of glass reactor and pumping argon (optimum pressure range is 53–80 KPa) into it. After filling the reactor with Argon, ablation process is carried out by focusing the laser pulses on the front face of graphite rod, and synchronously, the graphite rod is rotated at the rate of 20 rpm. After the graphite rod was consumed totally, the reactor tube wall would be covered with soot. The produced soot can be collected either using vacuum pump as a soot collector, or removing the glass reactor tube [76].

*4.5.2. Combustion reactor*

As described in Section 3.5 the process begins with pumping all the air out of reactor space, followed by injection of a premixed fuel consisting of oxygen, a light weight or straight-chained hydrocarbon (mostly benzene), an inert gas (commonly argon), and a photo-sensitizer substance such as $SF_6$ (as an energy transfer agent). Then the premixed fuel is ignited using laser radiation which creates a premixed flat flame as described in Section 2.5 The burner platform height is adjusted in a way that there is a short distance between tip of the flame and soot collector pump. While running fuel combustion inside the reactor, a pump placed on top of the premixed flat flame tip collects the soot [60].

## 4.6. Soot purification process

The purification process is the same as arc-discharge and combustion method.

# 5. Synthesis of fullerene by microwave

## 5.1. Synthesis process initials

This method uses microwave energy as heat source due to several microwave energy conveniences which can be cited for reaction time reduction, elimination of temperature gradients, superheating of solvents, and selective heating of precursors or catalysts [77–81]. One of the main advantages of using microwave energy as a heat source in fullerene production process is that microwave energy is not transferred by convection, conduction, or radiation. Unlike combustion method, this helps us to achieve homogeneous heating of precursors and also not damaging the reactor space.

Other industrial-oriented benefit of using microwave energy like heat source is the ability of running fullerene production process as a flexible manufacturing system, since the production process can be carried out inside an ordinary microwave synthesis device which is available in nearly all of the chemical research laboratories [82–84].

### 5.2. Fullerene generator system

The generator used in production process consists of a microwave synthesis device as heat source, active air cooling system inlets and outlets (compressed air mostly), and a borosilicate/$SiO_2$ microwave vial, $SiO_2$ vial is used if the temperature is higher than 800 °C, containing graphite powder as reactor space [85]. The generator system is shown in Fig. 4.

### 5.3. Carbon source

Graphite powder with particle size of μm order is utilized as carbon source, but carbon purified coal powder can also be used as carbon source. There is another microwave fullerene generator type which uses chloroform ($CHCl_3$) as carbon source, but due to similarity of utilized fullerene generator structure to combustion fullerene generator system, we will not describe it [85].

### 5.4. Neutralized environment

Unlike other methods described before, there is no need for neutralizing the reactor environment using a noble gas or by vacuuming all the air out of reactor space because microwave energy will not transfer by convection, conduction, or radiation, and this will help prevent any damage caused to reactor space by heat.

### 5.5. Production process explanation

The process of fullerene production using microwave synthesis starts with filling up a borosilicate/$SiO_2$ microwave vial with carbon source, mainly purified matrices of graphite, and placing the vial into a microwave synthesis device with operating frequency of 2.45 GHz corresponding to wavelength of 12.2 cm. Then, the microwave energy and active cooling using compressed air are applied to the graphite sample synchronously; active air cooling system is applied on the vial containing graphite sample. After these processes, a soot sample containing fullerenes is produced inside the vial [82,85].

### 5.6. Soot purification process

Soot purification can be carried out either by traditional or Soxhlet method. The soot can also be purified using toluene and ultrasonic bath (constant ultrasonic pulse).

## 6. Soot purification methods

As it was described earlier, all of the fullerene preparation methods produce unpurified soot containing different kinds of fullerenes, mostly C60 and other giant fullerenes such as C70, C84, C96, etc., and other carbonic nano-structures, i.e. carbon nano-tubes, nano-onions, etc. To obtain fullerene from soot sample, it should be purified. In order to purify soot sample, there are certain purification methods that can be employed such as traditional, and Soxhlet method. In the subsequent sections, soot purification methods mentioned earlier are described.

### 6.1. Traditional soot purification process

The reason that this method is called traditional is that it was the first method applied to purify fullerene containing soot sample. The purification process is carried out by washing the soot sample using toluene or benzene, and allowing a period of time to soak up. As it appears, this method will take some time to produce purified fullerene sample from soot. This method has a production yield range from moderate to low [86].

### 6.2. Soxhlet soot purification process

This method is carried out using a simple Soxhlet extractor system in which different kinds of straight-chained hydrocarbons such as hexane or heptane are used to extract different hids of fullerenes. Applying this method will lead to high extraction yields because a unique solvent is used to extract each type of fullerene (toluene for C60, hexane, heptane, etc. for giant fullerenes such as C70, C84, C96, etc.) [53,87].

There are other newly introduced purification methods, such as active carbon filtering [88], which could be industrialized in the future.

## 7. Discussion

As cited before, there are numerous fullerene synthesis methods which are divided in to two main divisions as chemical synthesis, and industrial-oriented synthesis. Also, scientists have introduced different synthesis methods and the formation mechanism of fullerenes [89–92]. That is the reason why all of the industrial-oriented methods produce fullerene as a side product along with producing other carbonic nanostructures such as carbon nano-tubes, nano-onions etc [93]. This issue has another effect on every fullerene synthesis method which is overall production yield, and this will in turn prevent new industrial fullerene synthesis methods from being introduced. There is a logical way of looking at this problem without introducing new synthesis methods, which is trying to improve vital production parameters of each process, since overall production yields among all of the methods are approximately in similar ranges, and improvement of parameters are more economically justifiable than introduction of new fullerene synthesis methods. Anyway, there are synthesis methods which will enable us to reach higher yields than other methods after vital improvements proposed to vital production parameters [94].

In next sections, at first vital production parameters, i.e. parameters having direct effect on overall production yields which are uniquely for a specific fullerene synthesis method, for each method are discussed and improvements for parameters are proposed. Then, vital common production parameters which are energy source, carbon source, reactor conditions including availability, reactor building costs, and maintenance, suitable purification methods, and green production levels for each method are discussed and compared with other methods in an effort to introduce the most industrially and economically justifiable method of fullerene production.

### 7.1. Vital and unique production parameters for each method

#### 7.1.1. Arc-discharge method

Vital production parameters for arc-discharge method are gap between electrodes, gas flow, low gas pressure, and high temperature. One of the signs of proper production conditions is obtaining a bright-coloured plasma. To obtain such plasma, there should be enough gap between electrodes. There is a range of distance between electrode tips in which the process of graphite vaporization starts, but not all the distances starting vaporization process are optimal for production; the only ranges of distance between electrode tips which are optimal are those which produce a bright-coloured plasma inside the reactor space. Of course, the optimal range of electrode gaps is unique for each arc-discharge reactor because of different shapes and dimensions of each reactor. However, by varying the distance between electrode tips, optimal distance producing bright-coloured plasma for each and every arc-discharge reactor can be determined [95].

Gas flow, on the other hand has a direct effect on production yields, because low velocity of gas can ensure completion of the carbonic structures which are similar to fullerenes that have incomplete structure, and will transform into fullerene if the missing carbon atoms are attached. As a conclusion, high velocity of gas will cause low production yields or increase of incomplete carbonic structures which are similar to fullerenes in the sample produced soot [96].

As we described above, to reach high yields of production, gas velocity should be low enough to ensure completion of carbonic structure similar to fullerenes. Since the temperature inside the reactor space is very high and this will cause high gas velocity, the gas pressure should be low enough to ensure low gas flow while reaching to very high temperatures. The suitable gas pressure can be determined by measuring the temperature and using a proper ideal gas equation. Also, the rate gas pressure is different for each and every arc-discharge reactor because of differences in size and dimensions [97].

The main vital production parameter is of course reaching very high temperatures, which will grant proper vaporization of electrodes. As we described earlier, applying direct current results in vaporization of just one electrode while alternative current will vaporize both electrodes. The direct current appears to be more efficient than the alternative because vaporization of both electrodes will lead to multiple stops in the middle of the process as the gap between tips of the electrodes will become distant soon enough to put an end to the whole arcing process. Applying a direct current of 20 V and 70 A will satisfy the need of reaching to very high temperatures and moving to higher currents such as 100 A [98–100].

There are other parameters which are not as important as the ones mentioned above, but they will increase production rate. These parameters are electrode thickness, electrode moving mechanism, moving an electrode forward as it is being consumed in the process to maintain suitable gap between tips of the electrodes which leads to higher production yields, and covering the internal reactor surface with a carbon source such as graphite to ensure completion of incomplete carbonic nano-structures which are similar to fullerenes [98].

#### *7.1.2. Combustion method*

Vital production parameters of combustion method are height of burner platform, vacuuming all the contents of reactor space, and very high temperature.

Burner platform height determines the height of premixed flat flame. Properly adjusted height for premixed flat flame results in proper fuel combustion conditions which grant incomplete fuel combustion leading to HTLP soot production with high temperature low pressure. The burner platform height will also prevent reactor walls from melting down. The optimum range of height for burner platform is of course different for each reactor due to differences in size and dimensions, but it can be obtained by varying the height and controlling it through observation window located on the outside of reactor space [58,101].

The main difference between arc-discharge and combustion reactor spaces is that arcing reactor is filled with a low pressure noble gas such as helium or argon, but combustion reactor is vacuumed all the way down instead. The main reason for vacuuming all the contents (air) out of reactor space in combustion method is that the temperature rises to very high peaks which will cause damages to reactor space. In order to prevent that, the neutralized environment should be as low thermally conductive as possible [102].

The last important production parameter having direct effect on fullerene production yields using combustion method is very high temperature [103]. In order to grant high temperatures, most proper premixed fuel should be used to create a high temperature premixed flat flame. Of course, any light weight or straight-chained hydrocarbon can be utilized to create a premixed fuel, but benzene appears to be the most convenient hydrocarbon to create a premixed fuel. The most important issue with selection of a hydrocarbon as a part of premixed fuel is that the selected hydrocarbon should meet two criteria which are high burning temperatures, and high rates of incomplete combustion which creates soot containing carbonic nano-structures, as well as different kinds fullerenes [104].

#### *7.1.3. Laser synthesis method*

*7.1.3.1. Vaporizer reactor.* Vital production parameters of laser reactor (vaporizer type) are low gas flow, and very high temperature. Since heating a gas under constant volume will raise its velocity and pressure, the noble gas, mainly helium, injected inside vacuumed reactor space should have lowest possible pressure to grant low gas velocity after starting the vaporization process. As we described in Section 7.1.1 low gas flow will lead to higher production yields as it ensures completion of incomplete carbonic nano-structures which are similar to fullerene which is incomplete yet [96].

According to many scientific works, fullerene formation will take place under high temperatures; therefore, reaching very high temperatures is vital for production. Of course, this can be easily achieved while using laser as a heat source, because laser radiation can be adjusted to reach very high temperatures, and carbon source used in the process which is mainly graphite as a very good heat absorber and will reach high temperatures easily and just on a point which will not cause damages to reactor space [69].

*7.1.3.2. Combustion reactor.* Vital parameters for fullerene production by combustion of a premixed fuel using laser radiation are the use of a photo-sensitizer substance, collecting soot simultaneously, and reaching very high temperature.

Photo-sensitizer substance is used as an energy transfer agent to ensure homogeneous heat dispersion, and also get as much thermal energy as possible from laser radiation. Getting as much energy as possible from laser radiation is very important because it will induce reaching very high temperature which is the most important parameter of production in almost every synthesis method [66].

The soot and other contents produced by the generator having really high temperatures should be collected as soon as the combustion process for that portion of fuel is carried out, because the contents will cause damage to reactor space. And settling the contents inside the reactor space will lead to complete combustion which causes production of soot in undesirable amounts (low amounts of soot) [105].

Reaching very high temperatures is a vital parameter of fullerene production which is accomplishable using laser radiation and a photo-sensitizer substance simultaneously.

#### *7.1.4. Microwave method*

Important production parameters of microwave synthesis are vials used as reactor space, active air-cooling system, and very high temperature.

Mainly borosilicate vials are used as reactor space, unless the temperature is very high (higher than 800 °C). If the temperature is higher than 800 °C, borosilicate vials should be replaced with $SiO_2$ vials, as borosilicate will melt down under temperatures higher than 800 °C [82].

Active air-cooling system using compressed air keeps the temperature gap between inside and outside of the vial used as reactor space, which is an advantage of microwave synthesis, because it enables us to continue raising the temperature of carbon source without actually damaging reactor space and keep the production process running until all of the carbon source has transformed into soot containing fullerenes. Very high temperature, on the other hand, will ensure vaporization of all of carbon source to produce soot containing fullerene; however, the desirable temperature while running microwave synthesis process is lower than any other industrial-oriented fullerene synthesis method [106].

#### *7.1.5. Table of specific parameters*

In other sections, we described vital production parameters for each

**Table 2**
Vital production parameters of each synthesis method.

| Fullerene synthesis method | Vital production parameters |
|---|---|
| Arc-discharge | Gap between electrodes, Gas flow, Low gas pressure, High temperature |
| Combustion | Height of burner platform, Vacuuming all the contents of reactor space, Very high temperature |
| Laser vaporization | Low gas flow, Very high temperature |
| Laser combustion | Use of a photo-sensitizer substance, Collecting soot simultaneously, Reaching to very high temperature |
| Microwave | Vials used as reactor space, Active air-cooling system, Very high temperature |

synthesis method. All of the parameters are shown in Table 2 for quick reference.

### 7.2. Common vital production parameters

#### 7.2.1. Synthesis process energy source

There are different energy sources for each synthesis method which contain electricity, different hydrocarbons as fuel, laser radiation, and microwave energy. Some of the mentioned energy sources are clean and convenient, but among all the methods only fuel combustion-based methods use hydrocarbons as energy source which is not as clean as electricity, laser vaporization, and microwave energy [107].

#### 7.2.2. Carbon source

Proper soot containing fullerene can be produced either by vaporizing a carbon source such as graphite, and coal, or by incomplete combustion of hydrocarbons. As time passes, use of hydrocarbons derived from oil as fuel becomes more inconvenient due to production of CO and $CO_2$ after fuel combustion process which will pollute the environment which we are living in, also due to oil shortage in future, hydrocarbons will become more costly than present. From what discussed, we can conclude that using coal [108] or graphite [109] as carbon source is more economically and industrially justifiable than hydrocarbons derived from oil. Of course, bio-fuels such as ethanol as products of fermentation of plants are better options for combustion reactor fuel, but obtaining these fuels from plants by fermentation process is costly and will take much time.

#### 7.2.3. Reactor conditions

*7.2.3.1. Availability and building costs.* Building each reactor has its own issues, but some reactors are cheaper than the others, for instance, arc-discharge. Microwave reactors are easily built in any material science lab while laser ablation, both combustion and vaporizer reactors, and combustion reactors use higher technology and are not as easy to build as arc-discharge and microwave reactors. The reason for partial unavailability to build laser and combustion reactors is that burner platform of combustion reactor should be built very accurately because slight misdirection of premixed flat flame can cause damages to reactor walls (melting down the walls), and also Nd:YAG laser used in laser ablation method is costly to be provided by science labs as ordinary material in order to build laser ablation reactor [110].

*7.2.3.2. Maintenance.* After soot production, if the reactor parts are damaged, each reactor should be cleaned and repaired. Among all of the reactors, microwave reactor needs least maintenance because microwave oven is not used as reactor space. Instead, a borosilicate/$SiO_2$ vial is used to hold graphite or coal as carbon source. This not only will not damage the microwave oven, but also will keep it clean and even make repairing the reactor space easier because damaged vials can be replaced by new vials [110,111].

#### 7.2.4. Purification methods

As we described earlier, there are two major methods of soot purification including traditional method, and Soxhlet method. Traditional method is carried out by washing soot sample using toluene, benzene, and other hydrocarbons each separating a different kind of fullerene. Soxhlet method however is more convenient than traditional method because it will consume less hydrocarbons used as solvents, and also will take lesser time than traditional method as in traditional method soot sample takes much time to soak up completely in hydrocarbons used as solvents [53,86,87].

### 7.3. Final comparison

Final comparison of fullerene synthesis methods by production parameters is shown in Table 3.

## 8. Conclusions

In this review, we explained all of the fullerene preparation methods which are industrial-oriented. Then, we described conditions and issues of each method under which soot containing fullerene will be produced. Also, we described different soot purification methods, followed by comparing all the fullerene synthesis methods and arguing which method is the most industrial-oriented, and economically justifiable. Although there are numerous fullerene synthesis methods, it appears that microwave and arc-discharge methods are still most convenient methods of fullerene preparation. But the most convenient method that we propose will be microwave method which uses carbon purified coal powder like carbon source, $SiO_2$ vial like reactor space, and Soxhlet soot purification process. Nevertheless, arc-discharge is the next convenient method which can be optimized to reach very high yields by proper reactor design and consideration of vital production parameters.

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## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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**Table 3**
Final comparison of fullerene synthesis methods by production parameters.

| Synthesis Method | Energy source | Carbon source | Maintenance costs | Reactor building costs |
|---|---|---|---|---|
| Arc-discharge | Electricity (clean) | Graphite, Coal (environment friendly) | Moderate | Low |
| Combustion | Fuel combustion (not clean) | Light-weight/Straight-chained hydrocarbons (polluting the environment) | High | High |
| Laser combustion | Laser radiation (clean) | Light-weight/Straight-chained hydrocarbons (polluting the environment) | High | High |
| Laser vaporization | Laser radiation (clean) | Graphite, Coal (environment friendly) | High | High |
| Microwave | Microwave energy (clean) | Graphite, Coal (environment friendly) | Low | Low |

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