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Preparation of slurry with colloid mill and influence of its particle size on the direct coal liquefaction

The coal object was obtained from Jiangjunmiao in Xinjiang; the coal slurry was prepared with colloid mill by wet milling. Influence of grinded time on particle size of coal slurry was investigated. Particle size distribution was measured by Laster granulometer. The result showed that particle size distribution was 8000 ~ 10800 nm (2000 ~ 1000), 2400 ~ 2900 (6000 ~ 5000), 800 ~ 1200 nm (18750 ~ 12500) and 500 ~ 1250 nm (30000 ~ 12000) grinded at 1h, 2h, 3h, 4h, respectively. Results of the direct coal liquefaction carried out under low pressure showed that oil yield would decline with decreasing of coal particle size. With the same liquefaction condition, oil yields of 200 and 1340 samples were 75.24 % and 59.96 %, respectively. The oil yield of sample 1340 treated by ultrasonic treatment rose significantly to 80.04 % and increased almost 20 %.

Key words: colloid mill, particle size, direct liquefaction, oil yield.

The content of water in the composition of dry coal slurry needs to be less than 4 %, but in case of atmospheric air, it is over than 5 %. The content of water with 50 % in the wet coal prevents slurry to dust off from coal. In the case of wet coal slurry with the high oxidation degree, the diameter of coal decreases and area increases. These provide the possibility of mixing, transporting and storing. In conclusion, the wet coal slurry is widely used than dry coal slurry [1]. Usually there are two methods of direct liquefaction such as heating and adding of hydrogen. During the heating of coal particles, the heating rate, dissolution velocity and gassing phenomena are different [2, 3]. In addition, the different size of particles influence on adding of hydrogen. That is why the area of contact between hydrogen and coal slurry are different in the presence of catalyst [4]. In the end, it affects on oil separation degree. In this research the liquefaction of Jiangjunmiao coal is carried out using milling and high intensive ultrasound processing. The use of ultrasound processing provides better coal liquefaction [5]. Afterwards under the low pressure the influence of particle size on coal liquefaction was investigated [6].

1. Experimental part

1.1 *Types of coal*. The Jiangjunmiao coal coal. Dry coal 200.

1.2 *The conditions of coal liquefaction*. As initial experiments showed, in the case of 1.5:1 the liquid of coal does not flow, and in the case of 2.5:1 one flows. The frictional force between molecules in the granules of coal decreases, grinding time prolongs, as a result, it needs more energy. The temperature of grinding machine increases, coal oxidizes, tetralin vapors. The optimal ratio is 2:1.

The control of grinding degree of colloid mill.

Figure 1. JML-50 Colloid mill

Figure 2. Adjustable pointer of colloid mill

1.3 *The particle size of coal slurry*. The 0.2 g coal slurry and 13 % ethanol were put in retort and closed by glass and the particle size was measured [7].

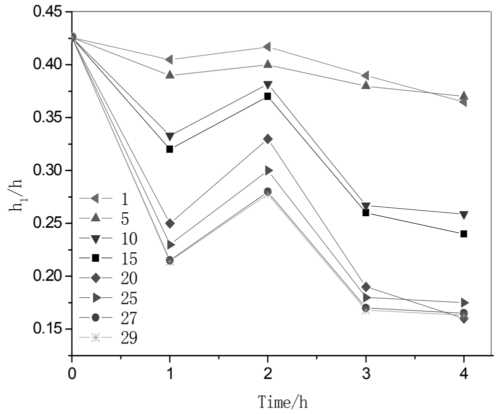


Figure 3. The connection between grinding time and liquid height

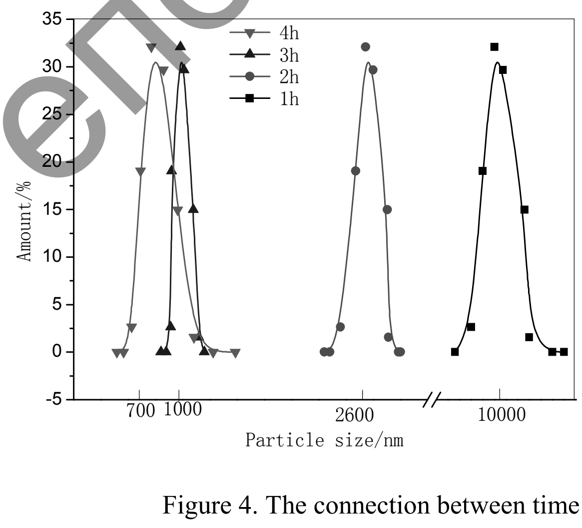
$$\eta_{Oil} = \frac{m_0 - m_1 - m_{solvent} - m_s}{m_{daf,\,coal}} \times 100\,\%,$$

m_0 — the liquefaction of solid after autoclave; m_1 — mass of ethanol; $m_{solvent}$ — mass of solvent; $m_{daf,\,coal}$ — mass of dry coal; m_s — mass of tetralin.

2. Conclusion and discussion

2.1 *The stable properties of liquid coal*. The control indicator of colloid mill was 29, 27, 25, 20, 15, 10, 5 and 1. The grinding time depending on liquid height was 2h>1h>3h> 4h, because the coal slurry decreased and agglomerated, as a result, the particle size increased. The height of division layer increased. During the 3h and 4h the size of agglomerated coal slurry increased and it was subjected to frictional force due to prolongation of grinding time. As the power of frictional force become more than agglomerate force, the size of coal slurry decreases, the height of division layer between coal slurry and solvent decreases too, stability increased. When time was about 1h, the height of division slurry decreased. When indicator showed 27 and 29, the coal liquid stabilized. At that time the particles of coal slurry decreased and the coal liquid stabilized [8, 9]. In the case of 30 two layers contacted with each other and experiment did not carry out. To provide the best conditions of experiments it is necessary to keep indicator at 27 [10, 11].

2.2 *The particle size distribution of coal liquid*. In the Figure 4 the distribution of coal slurry is shown after grinding time at 1h, 2h, 3h, 4h. It is represented that if the grinding time prolongs, the coal particle size will become smaller. During the 1h, 2h, 3h, 4h the distribution area of particles were 8000 ~ 10800 nm (2000 ~ 1000), 2400 ~ 2900 (6000 ~ 5000), 800 ~ 1200 nm (18750 ~ 12500) and 500 ~ 1250 nm (30000 ~ 12000), respectively.



Time, h	Mesh	Average particle size, μm
1	1340	10
2	5000	2.6
3	15000	1
4	20134	0.745

Figure 4. The connection between time and average size of coal slurry

During 1h, 3h and 4h the coal liquid is stable depending on layer of one, but 3h and 4h needs more energy, that is why the grinding time prolongs. The increase of temperature is undesirable for coal and equipment. That is why the experiment was carried out at 1h. In the end of experiment, the hydrogen was added under low pressure.

2.3 *The use of electric glass scanner during the work with colloid mill*. The results of electric glass scanner before and after colloid mill are shown in the Figure 5. In the Figure 5*a* the particle size of coal is large and shining. When particle of coal decreases, their size will be different due to various contents of minerals (Fig. 5*b*). In the definite interval of time, the coal size decreases and the minerals restore their state. The decreasing of coal size has influence on division of coal slurry and increase of surface area; all of these provide liquefaction of coal.

a — the electric glass scanner of coal before colloid mill

b — the electric glass scanner of coal with grinded at 1h (enlarged 200 times)

Figure 5. The electric glass scanner of coal slurry before and after colloid mill

2.4 *Investigation of changes in temperature*. Figure 6 represents the changes in temperature of coal before and after grinding. After grinding at 71.78c^0 coal lost its mass, because water is removed from one. The water of coal is more than one of grinded coal liquid which is dried by tetralin before measuring. At 167 ºC grinded coal liquid lost its mass for the second time due to removal of tetralin from coal. At 351–458 ºC the main grinding process took place and temperature interval is characterized as high point of mass loss. That is why after coal liquefaction it is necessary to remove the tetralin before measuring of the oil yield.

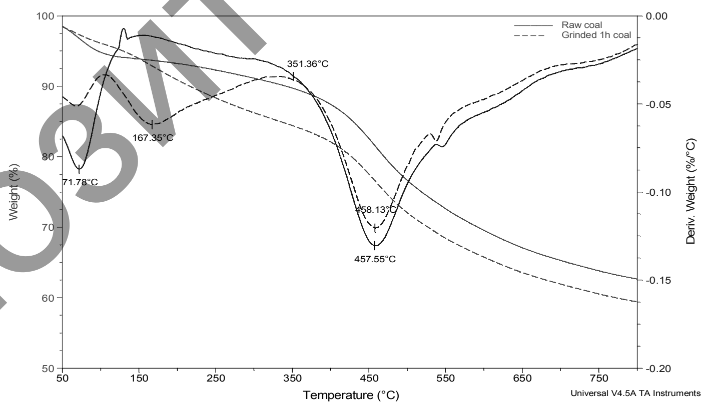


Figure 6. Comparison of TG-DTG curve on original coal and coal sample 1340 grinded at 1h

2.5 *The discussion of FT-IR spectroscopy*. The Figure 7 shows the FT- IR spectra of coal before and after grinding. It is established that the structure of two different coal is the same. After grinding, the strength of absorption is changed. The strength of laser absorption at 3400 cm^{-1}, 2920 cm^{-1}, 2310 cm^{-1}, 1600 cm^{-1} and 600 cm^{-1} for types of coal is the same. During grinding at 1h the laser absorption of benzene is more than one of original coal. This has a negative influence on liquefaction of coal.

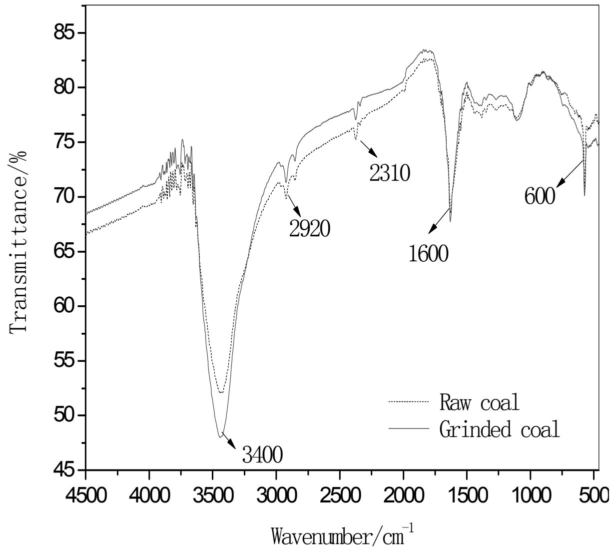


Figure 7. FT-IR spectra of original coal and grinded 1h coal

2.6 *The results of liquefaction of coal*. The results of direct liquefaction of coals 200 and 1340 by adding of hydrogen and using of colloid mill are shown in the Table.

T a b l e

The results of liquefaction of coal

Production rate	Oil ,%	As + Ps, %	Conv, %
Original coal (200)	75.24	9.71	92.96
During grinding at1h and without use of high intensive ultrasound	59.96	23.67	68.94
Grinding at1h and with use of high intensive ultrasound	80.04	7.58	95.15

It is shown from Table that the oil yield after grinding at 1h is 80.04 % which is more about 4.8 times than original coal. The exchange degree is 95,15 % which is more about 2.19 % than original coal. Without ultrasound processing oil yield is 59,96 % which is less about 12.2 % than original coal. Literature analysis shows that after grinding the coal particle size decreases, area increase, as a result, these provide to add the hydrogen. In addition, chemical properties of coal are changed and transformation of coal to oil is improved. As experiments showed, the agglomeration phenomena takes place with decreasing of coal slurry size. The high intensive ultrasound processing has a positive impact on liquidation of coal.

3. Conclusion

If the control indicator is at any points, the height of coal layers will be 2h > 1h > 3h > 4h due to the division layers. At 1h, 3h and 4h the coal slurry is in powder and the coal liquid is stable. During the 1h, the height between layers is the least at indication of 27 and the highest at indication of 1. That is why at indication of 27 the coal slurry size is the smallest and coal liquid is the most stable.

Based on laser measurements of coal liquid, the particle size of coal slurry decreases when coal is grinded. At 1h, 2h, 3h, 4h the division of coal sluryy is in interval at 8000 ~ 10800 nm (2000 ~ 1000), 2400 ~ 2900 (6000 ~ 5000), 800 ~ 1200 nm (18750 ~ 12500) and 500 ~ 1250 nm (30000 ~ 12000).

From graph of coal liquidation during grinding at1h: after colloid mill the coal size decreases, but distribution is different. It is shown from TG-DTG curve that after drying of coal slurry there is a tetralin in the content of coal before liquefaction, that is why at the end of experiments it is necessary to remove the solvent before measuring of oil yield. It is established from IR curve that there is an oxygen loss and increase of benzene rings during the coal grinding, these provide transformation of coal to liquid.

From experiment of liquefation of original coal: The oil yield increased 4.8 times using high intensive ultrasound processing. Without high intensive ultrasound processing the oil yield decreased on 15,2 %. That is why, the use of high intensive ultrasound processing is important in liquefaction of coal.

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Көмір суспензияларын коллоидтық диірмен арқылы дайындау және суспензия бөлшектері мөлшерінің көмірді тікелей сұйылту үрдісіне әсері

Зерттеу нысаны синьцзяндық көмір болып табылады, көмір суспензиялары коллоидтық диірмен арқылы дайындалды. Үгіту уақытының көмір суспензия бөлшектерінің мөлшеріне әсері зерттелді. Гранулометриялық сараптама Laster гранулометрі арқылы жасалды. Алынған нәтижелер бойынша, 1, 2, 3, 4 сағат кезіндегі бөлшектер мөлшерінің таралуы келесідей: 8000 ~ 10800 нм (2000 ~ 1000); 2400 ~ 2900 (6000 ~ 5000); 800 ~ 1200 nm (18750 ~ 12500) және 500 ~ 1250 нм (30000 ~ 12000). Төменгі қысымда жүргізілген көмірдің сұйылту нәтижесінде бөлшектердің мөлшері кішірейген сайын мұнайдың шығымы азаятындығы байқалды. Жоғарыда аталған шарттарда 200 және 1340 үлгілері майдың шығымы бойынша 75,24 және 59,96 % құрады. Ал 1340 үлгісін ультрадыбыспен өңдегенде май шығымы 80,04 % жетті, яғни 20 % артты.

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Приготовление угольных суспензий с помощью коллоидной мельницы и влияние частиц суспензий на прямое сжижение угля

В качестве объекта исследования использовался синьцзянский уголь, угольные суспензии готовили с помощью коллоидной мельницы мокрого помола. Исследовано влияние времени измельчения на размер частиц угольной суспензии. Гранулометрический анализ проводился Laster гранулометром. По полученным результатам, распределение размер частиц следующие: 8000 ~ 10800 нм (2000 ~ 1000), 2400 ~ 2900 (6000 ~ 5000), 800 ~ 1200 nm (18750 ~ 12500) и 500 ~ 1250 нм (30000 ~ 12000) при 1, 2, 3, 4 ч соответственно. Результаты прямого сжижения угля при низком давлении показали, что выход нефти снижается с уменьшением размеров частиц. При таких же условиях выходы масел образцов 200 и 1340 составляют соответственно 75,24 % и 59,96 %. При ультразвуковой обработке образца 1340 выход масла составил до 80,04 %, т.е. увеличился почти на 20 %.