

SHINAGAWA TECHNICAL REPORT

コークス炉装入孔用スラリータイプシール材

Slurry-type Sealing Compound for Coal Charging Holes in Coke Oven

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品川技報

製品紹介 PRODUCTS

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要 旨

コークス炉では炉頂の装入孔枠と蓋の隙間からの炉内ガスリークの防止を目的に、粉末と水を混合して調整したスラリーが注入される。しかし、スラリーの調整が不十分だとシール不十分、注入時の排出不良、スラリー搬送時の圧送不良等の問題が生じる。これに対してスラリータイプのシール材XX-12W-1を開発した。これは粉末タイプの従来品と異なり製造工場で作成したスラリーとして出荷されるため現場での煩雑なスラリー調整が不要となる。スラリーは製造工場で品質管理されており、現場におけるスラリーの調整不良に起因する各種問題を生じ難い。中でもシール性は従来品より向上しており、新開発のスラリータイプシール材XX-12-W-1を使用することでシール性に起因する問題を大幅に低減することが可能となる。

Abstract

In order to prevent internal coke oven gas leakage out of the thin gaps between the coal charging hole frames and the charging hole lids on the top of coke oven, the gaps are filled with a slurry that is prepared by mixing sealing powders with water. Due to inadequate preparation of the slurry, operational problems such as insufficient sealing, drainage failure, or malfunction during pressure-feeding operation of the slurry, are sporadically observed. The novel slurry-type sealing compound XX-12-W-1 was developed to eliminate these problems. Unlike conventional powder-type sealing products, which require on-site slurry preparation, XX-12-W-1 is produced completely in our manufacturing plant, from mixing to container packaging. It can be used without the burdensome on-site preparation. Since this novel slurry-type sealing compound is produced under precise quality control, it's less likely to cause the above-mentioned problems mainly caused by inadequate on-site slurry preparation. Especially, the sealing capability was significantly improved compared to conventional products, XX-12-W-1 remarkably reduces operational problems caused by insufficient sealing capability of sealing compounds.

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1. コークス炉装入孔用シール材

コークス炉は石炭を乾留してコークスとガスを製造する設備で、Fig.1に示すように石炭は炉頂に設けられた装炭車から装入孔に投入され約1日掛けて乾留することでコークスを製造する。得られたコークスは側壁のドアを外して押し出し機により水平方向に排出される。石炭の乾溜中、炉内ではピッチガスが発生し、これを捕集し有機化学製品の原料に利用される。しかしピッチガスは環境や人体に有害であり、炉外に放出されないように入荷孔枠と蓋の間にできた1 mm未満程度の隙間にシール材を注ぎ込んで密封することが一般的である (Fig.2)。

シール材はスラリー状で、炉頂の装炭車に設置されたタンクに貯蔵されており、操業では、蓋開

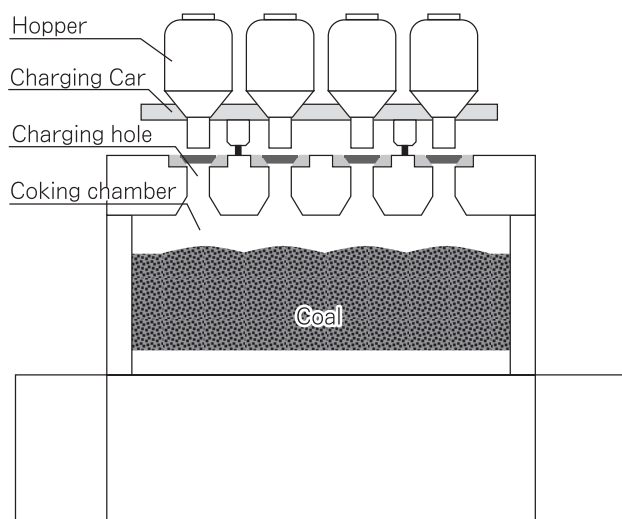


Fig. 1 Schematic illustration of major configurations of coke chambers in the coke oven.

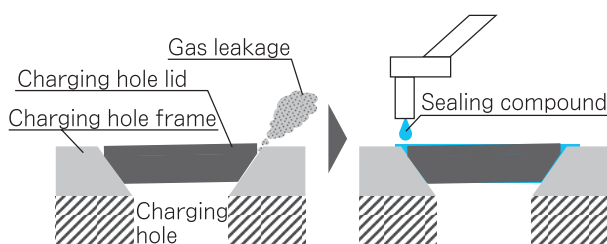


Fig. 2 Schematic illustration of coal charging hole.

1. Sealing Compound for Coal Charging Hole

The coke-oven is one of the carbonization furnaces that produce coke from coking coal. As shown in Fig. 1, the charging car charges the coal into the coking chamber from the coal charging hole situated on the top of the furnace and dry distills it for about a day. When the dry distillation step is completed, the coke is discharged horizontally by the coke pusher car, from a previously opened door installed in the side wall. The pitch gas generated in coking chambers during dry distillation of coal is collected as a by-product in order to use it as raw material for organic chemical products. However, pitch gas is considered to be hazardous to both atmosphere and humans. As shown in Fig. 2, pitch gas emission into the atmosphere is usually suppressed by filling a sealing slurry into the less than 1 mm gap between the charging hole frame and charging hole lid.

The sealing slurry is stored in a storage tank situated on the coal charging car, installed on the top of the furnace. The coal charging operation is conducted automatically, it usually begins with lifting the lid to open the charging hole, coal charging into the coking chamber, closing the charging hole, and ends with pouring the sealing compound to fill the thin gap between the charging hole frame and the lid. This sealing slurry is usually prepared by operators by mixing the sealing powder with water on the ground level, then the slurry is pressure-fed to the storage tank on the coal charging car on the top of the furnace.

2. Malfunctions of the Conventional Sealing Compounds

1) Insufficient sealing

Despite pouring an adequate quantity of the sealant, it is occasionally observed that the gas leakage fails to be eliminated, as the sealing

放、石炭装入、蓋閉鎖、シール材スラリー注入、の一連の動作が自動で行われる。シール材スラリーは地上の作業場で作業者が粉末と水をよく混合することで得られ、炉頂の装炭車までポンプで圧送される。

2. シール材を使用する際に発生する問題

1) シール不十分

所定量のシール材を注入したにも関わらずガスリークが止まらない場合がある。これはスラリーが装入孔枠と蓋の隙間に留まらず炉内に流下するため、スラリー粘度が低すぎると生じる。この場合、ガスリークを止めるために作業者は炉頂に上がり、通常より高濃度のスラリーを作製し装入孔と蓋の間に手動で注入しなければならない。

2) 排出不良

排出不良は装炭車からスラリーが排出されない現象で、スラリーが不安定な場合、粉末が凝集して、粉末がタンク内に固着することで生じる。この場合も人力による装入孔のシールが必要で、タンクからスラリーの抜き取り清掃作業も発生する。

3) 固着

装入孔の蓋は炉の整備や点検時に人力で開閉される。そのとき装入孔と蓋が強固に固着していると蓋の開放が重労働となる。これはシール材の接着力が高いと起こりやすい。

4) 圧送不良

調整したスラリーを炉頂に圧送できない圧送不良は、スラリーの粘度が高すぎるときに起こる。

5) 装入孔枠と蓋の摩耗

装入孔の蓋は開閉操作により擦れ合い摩耗する。シール材が硬いと研磨剤のように働いて装入孔と蓋の摩耗が助長される。

3. 新開発スラリータイプシール材 XX-12-W-1

前述の問題に対してスラリータイプのシール材XX-12W-1を開発した。これは当社粉末タイプの従来品であるMS-9-SRをベースに大幅な改良を加えたものである。従来品MS-9-SR-1は固着し難く軟らかい原料として炭酸カルシウムを用い、

compound slips into the coking chamber without filling the gap between the charging hole frame and the charging hole lid. Such problem could be due to the low viscosity of the sealing compound. In order to prevent the mentioned gas leakage, a sealing compound with higher viscosity is prepared on the top of the furnace and applied manually by operators.

2) Drainage failure of the sealing compound

When the sealing compound is unstable, the sealing powder tends to aggregate in the slurry, which leads to an accumulation of the sealant on the inner wall of the storage tank, resulting in a drainage failure of the slurry. In such cases, operators are required then, to manually apply the sealing compound in the gap between the charging hole frame and the charging hole lid, pumping out the sealing slurry and cleaning the tank.

3) Excessive adhesion

During maintenance or the inspection of the coke oven, the charging hole lid is manually lifted by operators. Therefore, excessive adhesion between the lid and the hole frame demands an intense physical effort from operators. Such an undesirable characteristic is caused by the excessive adhesion strength of the sealing compound.

4) Malfunction in pressure-feeding of the sealing compound

The high viscosity of the sealing compound becomes problematic during the pressure feeding operation, as the slurry cannot be properly pressure-fed to the storage tank at the top of the furnace.

5) The wearing out of coal charging hole frame and lid

The charging hole frame and the charging hole lid gradually wear out due to friction. This friction is a result of the repeated opening and closing of the lid. Furthermore, if the sealing compound is harder than the frame or the lid, its effect is similar to an abrasive.

シール材に適した粒度に調整したもので、そのスラリーはシール性に優れる。しかしスラリーの分散安定性が低く、スラリー調整後長期間経過すると分散が不安定になり、粘度にバラツキが生じ、前述の諸問題が生じる可能性があった。

XX-12-W-1は、MS-9-SRに特殊な安定化剤と新開発の製法からなる分散安定化技術(特許出願中)を適用したもので、分散安定性に非常に優れるため、早期分離に起因する排出不良を生じ難い。

そのため製造工場でスラリー化され、一斗缶や専用容器に入れて出荷される。現場ではこれをタンクに移すだけでよく、煩雑なスラリー調整作業が不要となる。加えて、製造工場では正確に室温で $45\sim 55\text{mPa}\cdot\text{s}$ の粘度になるよう品質管理されているため、現場でスラリー調整する際に生じやすい粘度のバラつきがなく圧送不良が生じ難い。

さらに最も重要な機能であるシール性が、適正に調整された従来品のスラリーよりも向上している。この新開発のスラリータイプシール材XX-12-W-1を使用することでシール性に起因する問題を大幅に低減することが可能となる。

以下にスラリータイプシール材XX-12W-1の

Thus, it aggravates the wearing out previously mentioned.

3. Novel Slurry-type Sealing Compound XX-12-W-1

XX-12-W-1 was developed to solve the above problems. This is a significantly improved product based on MS-9-SR, the conventional sealing compound. This powder-type sealant is calcium carbonate based, with relatively soft particles and appropriate adhesion strength. Thanks to its optimized grain distribution, this conventional powder-type sealant has an excellent sealing capability. However, due to its low dispersion stability, when the sealing slurry is left a considerable time after being prepared, its viscosity tends to change, this poor dispersion stability is the major cause of the above-mentioned problems of the sealing compound.

XX-12-W-1 was developed by combining MS-9-SR with a special dispersion stabilizer and a newly developed dispersion stabilizing technique (patent pending). The novel XX-12-W-1 has

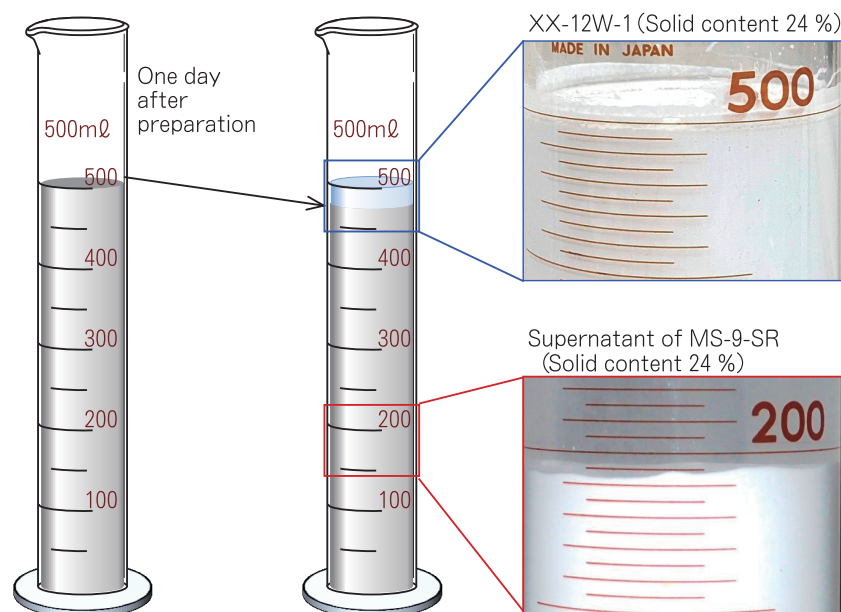


Fig. 3 Supernatant (clear liquid) formed in both, XX-12-W-1 slurry-type sealing compound, and MS-9-SR powder-type sealing compound, one day after preparation.

特性評価結果を示す。

1) 分散安定性

Fig.3に当社従来シール材MS-9-SRの水スラリー（固形分24%）とXX-12W-1（固形分24%）を500mlメスシリンダー内で1日静置した時の上澄み液の様子を示す。MS-9-SRは1日で上澄み液量が308ml（61.6vol%）になったことに対し、XX-12W-1は2.5ml（0.5vol%）とほとんど上澄み液が発生しなかった。

Fig.4にXX-12W-1を500mlメスシリンダー内で5週間静置した時の上澄みの様子を、Fig.5にXX-12W-1静置期間と上澄み量の関係を示す。XX-12W-1は5週間静置後において上澄み発生量が21ml（4.2vol%）と、MS-9-SRの水スラリーの1日

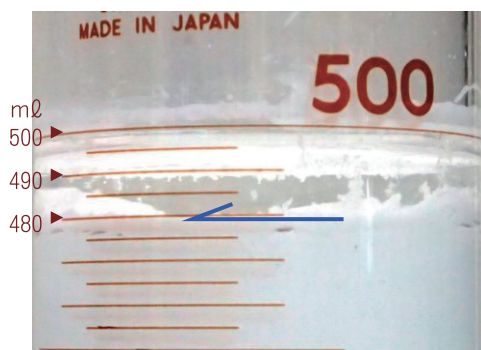


Fig. 4 Supernatant (clear liquid) formed in slurry-type XX-12-W-1 sealing compound, after being allowed to settle and stabilize for 5 weeks.

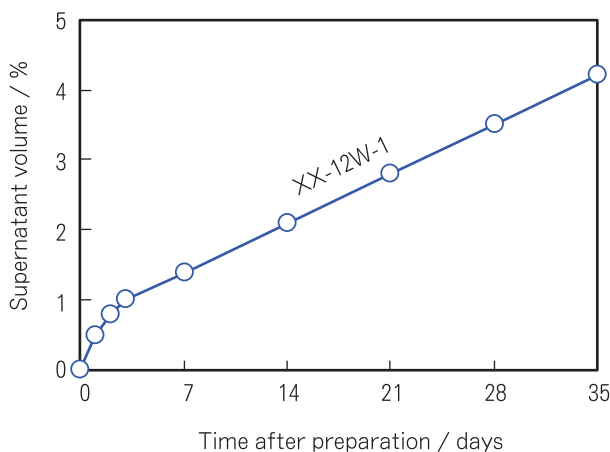


Fig. 5 Relation between after-preparation time and supernatant volume formed in XX-12-W-1.

high dispersion stability, therefore, the drainage failure caused by early destabilization of the slurry is less likely to occur.

After being prepared in our manufacturing plant, the slurry-type compound is delivered in any desired container, including an eighteen litre drum. On site, the slurry is simply transferred to the tank, thus eliminating the burdensome on-site slurry preparation. Furthermore, as a result of precise quality control, the steady viscosity of XX-12-W-1 is adjusted to the range of 40~55 mPa·s at room temperature, thus preventing any pressure-feeding problems caused by viscosity fluctuation of the slurry.

The most important property of sealing compounds is their sealing capability. This novel developed product capability to ensure an adequate sealing, exceeds the conventional sealing compounds' even when they are properly mixed. Therefore, by using this newly developed XX-12-W-1, the problems caused by insufficient sealing are highly reduced.

The evaluation results of the main characteristics of the slurry-type sealing compound XX-12-W-1 are summarized below.

1) Dispersion stability

One day after being prepared, the conventional sealing compound MS-9-SR (solid content 24 wt%) showed a supernatant (clear liquid) of 308 ml (61.6 vol%). While the novel XX-12-W-1 (solid content 24 wt%) showed a slight supernatant (clear liquid) of 2.5 ml (0.5 vol%) as shown in Fig. 3

The slurry was poured into a 500 ml graduated cylinder and was allowed to settle and stabilize for 5 weeks. As shown in Fig. 4, XX-12-W-1 had a supernatant of 21 ml (4.2 vol%), which is one-tenth of MS-9-SR's supernatant volume formed in one day after preparation. As shown in Fig. 5, the supernatant volume of XX-12-W-1 increases in a gradual rate starting from day 6.

However, the dispersion stability could not

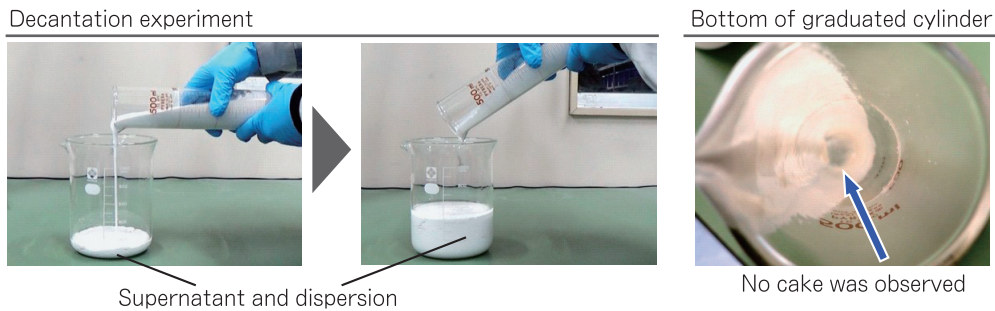


Fig. 6 Snapshots of decantation experiment concerning cake-formation of XX-12-W-1 after being allowed to settle and stabilize for 5 weeks in graduated cylinder.

静置後の10分の1程度であった。また、1週間以降の上澄み発生量の増加は緩やかであった。

しかし、ケーキ状の沈殿があると分散安定性に優れるとは言えないため、メスシリンダーを傾けて分散部を注意深く排出し、ケーキ状の沈殿の有無を確認した。Fig.6に5週間静置後のXX-12W-1をゆっくり排出したときの様子を示す。排出する時にやや濃厚に見える部分があったが全て排出され、ケーキ状の沈殿はなかった。さらに、排出したXX-12W-1を500mlメスシリンダーに戻し、倒置することで攪拌すると、一度の倒置で濃厚に見えた部分もなくなり、再分散しやすかった。

XX-12W-1は、500mlメスシリンダー内で5週間静置後でも上澄み発生量が比較的少なく、またケーキ状の沈殿も発生しなかった。これらよりXX-12W-1は分散安定性に優れることが示された。

これより、装炭車に設置されたタンク内で攪拌されていれば、タンク内固着の解消が期待できる。また、手作業によるシールの場合も再分散性に優れることから一斗缶のような小さな容器で保管しておき、シール不良時のみ容器を揺動して分散してから使用することが可能である。そのため、粉末製品から高濃度のスラリーを作製する手間がなくなり作業負荷の軽減が期待できる。

2) シール性

Fig.7にシール性評価の概要を示す。シール性の評価は装入孔栓材質で製作した2つのプレキャストブロックを300℃に温め、長さ170mm、幅1mm、奥行き49mmの隙間を作り、その隙間にシール材

be thought of as excellent if cake formation was observed in the bottom of the graduated cylinder. Therefore, XX-12-W-1 was carefully poured into another container as shown in Fig.6. A small amount of slightly thick slurry was observed at the beginning of pouring. However, after pouring out all the content in the container, no cake formation was observed at the bottom of the graduated cylinder. After pouring back XX-12-W-1 to the graduated cylinder, the slurry was stirred by inversion and the small previous thick part was successfully eliminated, which indicates that the slurry is easy to re-disperse.

After preparation, XX-12-W-1 was allowed to settle and stabilize for 5 weeks. The supernatant's volume was relatively small, and no cake formation was observed at the bottom of the graduated cylinder. Thus, it could be concluded that XX-12-W-1 has an excellent dispersion stability.

Based on the results of the detailed experiment mentioned above, the accumulation of the sealant on the inner wall of the storage tank is expected to be easily suppressed when the slurry is agitated in the tank on the charging car. Also, in the case of sealing failure, before manual sealing, the necessity to prepare a slurry with higher viscosity is eliminated, as the sealing compound could be stored in any container size, including an eighteen litre drum. Thus, the sealing slurry could be re-dispersed

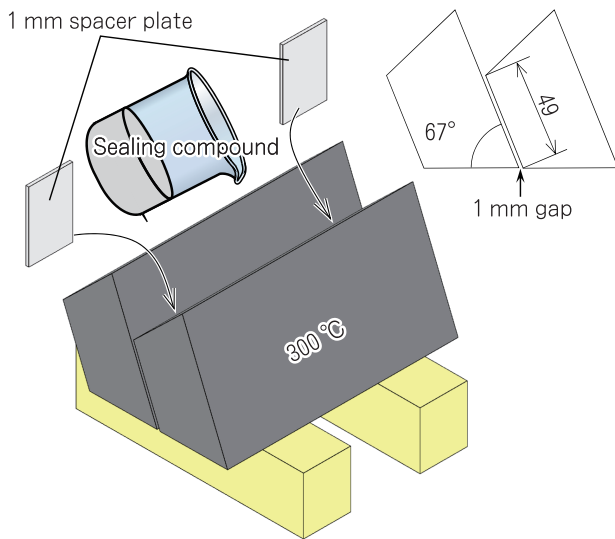


Fig. 7 Schematically illustrated evaluation method for sealing capability of sealing compounds.

200gを流し込むことで行った。シール材の流下が止まるまでの時間が短いほどシール性に優れるとした。

Fig.8にシール性評価の様子を、Fig.9にシール性評価後のシール面を示す。従来シール材MS-9-SRの水スラリー（固形分24%）は、シール材を全て流し込んでも隙間からの流下が止まることなく、シール面にはほとんど材料が残留していなかった。実機でのシール不十分は、このように隙間が大きすぎてシール材が流下して発生すると類推できる。これに対し、XX-12W-1は31秒で流下が止まり、シール面は材料で満たされていた。これより実機でもシール性不足によるシール不良は解消され则认为る。

easily by shaking the designed container.

2) Sealing capability

As schematically illustrated in Fig.7, the evaluation test for the sealing capability of the sealing compound was conducted as follows, 200 g of the sealing compound was poured in a gap of 1 mm width, 17 mm length and 49 mm depth, between the two precast blocks, heated to 300 °C, of the same material as the hole frame. The shorter time it took for the sealing slurry to stop leaking from the gap, the higher the sealing capability was considered to be.

Fig.8 shows several snapshots taken during the evaluation test of XX-12-W-1. Fig.9 shows the sealing surface of the precast blocks after the evaluation test. After pouring 200 g of the conventional MS-9-SR (solid content 24 %) in the gap, almost no sealing compound remained on the surface of the precast blocks, as the slurry kept on leaking through as shown on the left picture in Fig.9. It was assumed then, that an insufficient sealing of the coal charging hole might occur in the actual coke oven, as the conventional material will leak into the coking chambers through the relatively wide gap between the charging hole frame and the charging hole lid. Meanwhile in the case of slurry-type sealing compound XX-12-W-1, the slurry stopped leaking after 31 seconds and the surface area of the precast blocks was completely sealed with sufficient thickness as shown on the right side of Fig.9. Thus, novel slurry-type XX-12-W-1 will help to eliminate the insufficient

XX-12W-1



Fig. 8 Snapshots of sealing capability evaluation test.

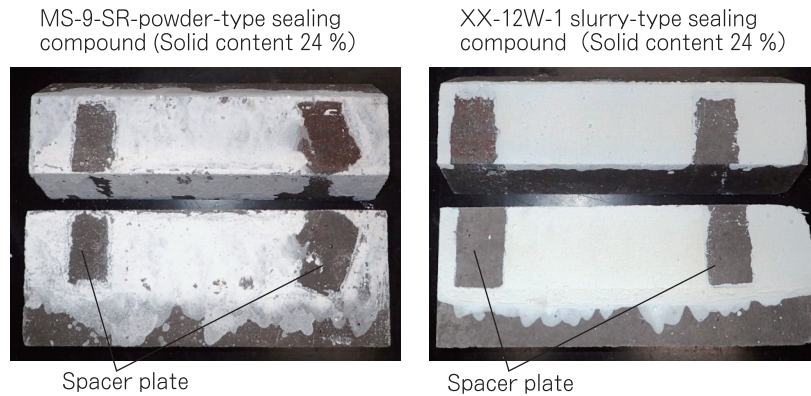


Fig. 9 Sealed surfaces of precast blocks after sealing capability evaluation test.

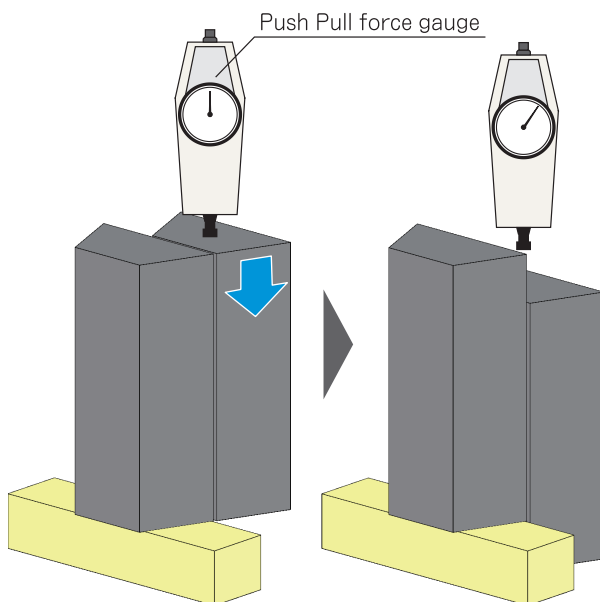


Fig. 10 Schematic illustration of adhesive strength evaluation method.

3) 剥離性

シーリング材の固着しにくさ、すなわち剥離性は接着強度で評価した。Fig.10に接着強度評価の概要を示す。接着したプレキャストブロックの片方の端面をプッシュプルゲージで剥がれるまで縦方向に押し、剥がれた時の荷重とプレキャストブロック重量と接着面積より接着強度を算出した。XX-12W-1の接着強度は4.8kPa (0.0048MPa)と小さく脱着に支障はないと考える。

sealing caused by the poor sealing capability of conventional sealing compounds.

3) Detachability

The detachability of the precast blocks was evaluated through their adhesiveness to the sealing compound as shown in Fig. 10. A push-pull force gauge was vertically attached to one of the precast blocks, then, this precast block was pushed down until it detached from the adhered surface area. The adhesive strength was then calculated by considering the weight of the precast block, the adhered surface area and the detachment force. The adhesive strength of XX-12-W-1 was calculated to be 4.8 kPa (0.0048 MPa), which is thought to be adequate to ensure easy opening of the coal charging hole in the coke-oven furnace.

4. Conclusion

A novel slurry-type sealing compound named XX-12-W-1 has been developed to be effective in reducing the internal coke oven gas leakage from the gaps between the coal charging holes and the coal charging lids. Unlike conventional powder-type sealing compounds, which require on-site slurry preparation, XX-12-W-1 is fully produced in our manufacturing plant, and undergoes precise quality control, to be delivered as a slurry sealant, thus eliminating the burden of the on-site slurry preparation and the

4. まとめ

コークスの炉装入孔枠と蓋の隙間からのガスリークの低減に効果的なスラリータイプシール材XX-12W-1を開発した。これは従来の現場でのスラリー調整が必要な粉末タイプ製品と異なり、製造工場で優れた品質管理の下スラリー化されて出荷されるため、現場での煩雑なスラリー調整が不要となり、粘度のバラツキに伴う圧送不良も起こさない。また分散安定性とシール性が従来製品より大幅に改善されている。これらより新たに開発したXX-12W-1を使用することで、タンク内への固着がなくなり、排出不良に伴うシール不良が解消されるだけでなく、コークス炉装入孔と蓋の隙間からのガスリークを大幅に低減することが可能となる。

malfunction in pressure feeding caused by the viscosity fluctuation of the sealing compounds. Furthermore, the dispersion stability and the sealing capability have been significantly improved compared to conventional products. It can be concluded then, that the accumulation of the sealant on the inner wall of the storage tank will be eliminated by using newly developed slurry-type sealing compound XX-12-W-1. This means that the insufficient sealing caused by the drainage failure of the slurry from the storage tank can be effectively suppressed. Moreover, it will markedly reduce the operational problems caused by insufficient sealing capability of the conventional sealing compounds.