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Abstract

The situation of availability of the good coking coal neither permits nowadays to plan a new charging blend carefully guite in time nor to charge it to the coke ovens for a long period. The availability of suitable quantities on the spot market often decides about the coals for the blend. Thus the operators have to face a situation of frequently changing coal basis. With respect to this situation every helpful means would be appreciated to receive rapid and reliable information about the influence of varied blend properties on coke quality or to optimize the blend concerning economical aspects. The semi-technical scale carbonization test ovens of DMT and the small-scale coking retort are well-known and have been using to monitor the carbonization behavior of individual coals and to optimize coking coal blends since several decades successfully. Nearly simultaneously a prediction model to estimate the coke quality has been developed based on significant coal data. Recent effort and achievement on the prediction model expand its use not only for the coke quality prediction, but to a coal blend optimization prediction model that includes the technical and economic constraints of coke oven operator. DMT will present on two practical cases how the equipment of the DMT cokemaking facility and the enhanced prediction model allows to optimize coal blends concerning coke quality, coke oven safety and economics.

Key words: Cokemaking; Coal blend optimization; Coke quality; Coke oven safety.

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1 INTRODUCTION

Semi-industrial carbonization tests are very valuable to test coking coal blends before charging such a blend into the industrial scale coke oven. Nowadays time saving and cost effective methods of pilot scale carbonization tests as well as mathematical models for the estimation of important coke properties from coal data are available to support semi-industrial scale coal testing. The very important coke quality characteristics CRI and CSR can reliably be calculated, even without major restrictions of the origin of the coal components. Despite from that a realistic confirmation by semi-industrial scale carbonization tests is recommended, since they deliver further valuable information for the coke plant operator concerning e.g. the swelling pressure. Also the conventional drum tests can be performed in order to receive the complete set of significant coke quality characteristics. Furthermore the combination of coke quality estimation by calculation models with the semi-industrial and pilot scale carbonization tests permits to receive supplementary information which could not be gained by applying only one of these methods.

2 TECHNICAL FACILITIES AND PREDICTION MODEL

2.1 Semi-Industrial Carbonization Test Ovens

DMT is operating the following two semi-industrial carbonization test ovens:

- Standard oven with fixed heating walls 450 mm chamber width and 250 kg coal capacity
- Movable wall oven 450 to 850 mm chamber width and 500 to 1000 kg coal capacity.

Both semi-industrial ovens are underfired with natural gas. The refractory brick work consists of Silica bricks and the heating flues have similar cross cut dimensions as those of the industrial scale oven. Therefore the heat transfer from the heating walls into the coal charge occurs in a very similar manner compared with the industrial scale plant. Thus an optimum simulation of the plant conditions is guaranteed.

Figure 1 displays a cross cut of the movable wall oven. The chamber width is the decisive dimension for the heat transfer from the heating walls to the coal charge and is simulated in the semi-industrial coke oven in the scale 1:1. Length and height of the semi-industrial coke oven chamber are reduced quite significantly: the distance between the door plugs is approx. 1,3 m and the charging height approx. 1,0 m. Besides from the possibility to vary the chamber width the movable wall oven has the advantage that the forces exerted to the movable wall during carbonization by the swelling pressure of the charge can directly be monitored via load cells.



Figure 1. Semi-industrial carbonization test oven with movable wall.

Under the premises that the operating conditions are exactly simulated concerning size distribution, moisture content, bulk density and heating flue temperature reliable results for the plant operator can be guaranteed with respect to the swelling behavior of the coal charge and the resulting coke quality^[1].

2.2 Pilot Scale Carbonization Test Oven

Compared with the semi-industrial coke oven the 10-kg-retort permits less time consuming investigations with limited quantities of coal (approx. 11 kg charging weight). Figure 2 contains the most important data of the carbonization retort. While the semi-industrial oven is always operated as close as possible to the plant conditions, the heating conditions of the electrically heated retort are always fixed standard conditions. After a coking time of approx. 4 hours a final coke temperature between 1030 and 1040°C is attained. Size distribution moisture content and bulk density are adjusted as given by the industrial scale plant. Despite from the fact that the course of carbonization differs in some details from the industrial scale oven the pilot scale carbonization delivers valuable results concerning swelling pressure and coke quality with respect to CRI and CSR. The formation of an entirely closed plastic zone (like an envelope) during carbonization is first of all very decisive for the good correlation of CRI and CSR values with the values to be expected at the plant but it also permits a reliable assessment of the swelling characteristics. On the one hand a gas pressure is generated within the plastic envelop which proved to be higher with increasing viscosity of the plastic mass. Thus conclusions can be made with respect to a possibly dangerous swelling behavior. On the other hand the raw gas is always forced to pass through the hot coke before it reaches the gas free space. This leads to a maximum of cracking reactions in the pore structure of the incandescent coke. Especially this fact is responsible for CRI and CSR values which are comparable with those of the industrial scale plant^[2,3].





Figure 2. Pilot scale carbonization retort (10-kg-retort).

2.3 Prediction Model

The statistical evaluation of approx. 150 semi-industrial carbonization tests have been the basis at DMT for the development of a calculation formula to estimate the coke quality indices CRI and CSR from available coal data.

The parameters involved in these equations are listed in Table 1. Only those have involved which proved to be significant from the statistical evaluation^[4,5].

Deremeter	Parameters considered in the calculation formula		
Farameter	CRI	CSR	
G value	+	+	
Mean reflectance R _m	+	+	
Vitrinite content	-	+	
Distribution of vitrinite reflectance	+	+	
Fe ₂ O ₃ content	+	+	
CaO content	+	+	
K ₂ O content	+	-	
P ₂ O ₅ content	+	+	
Bulk density	_	+	

Table 1. Parameters considered in the calculation formulas for CRI und CSR

A special ability of the calculation method has to be pointed out. Besides from the calculation of CRI and CSR supplementary valuable information can be received about the individual positive or negative influence of certain coal properties on CRI and CSR. An example of a coal which was decisively modified in its ash content and ash composition by different preparation techniques shall elucidate this ability and calculated as well as measured values shall be compared. The Table 2 shows some selected properties of the differently prepared coals. The two variations are characterized as R1 and R2. While coal R1 has a very low ash content of only 2.68% (mf) the ash content of coal R2 of 8.42% (mf) has to be considered as normal.



Table 2. Selected properties of coals R1 and R2

		Coal R1	Coal R2
Ash (mf)	%	2.68	8.42
Volatile matter content (mf)	%	24.8	24.0
Volatile matter content (maf)	%	25.5	26.2
G value		1.008	1.000
Mean reflectance R _m	%	1.15	1.14
Vitrinite content	Vol%	44	45
Bulk density, wet	kg/m³	880	880
Coal moisture	%	9.2	9.2

Table 3 displays the differences in ash composition. The high CaO content is conspicuous for both coals. The CaO content of R2 is already an extremely high value. The values for Fe₂O₃, K₂O and P₂O₅ reflect normal ranges.

Table 3. Chemical composition of the coal ash

		Coal R1	Coal R2
SiO ₂	%	44.0	44.0
AI_2O_3	%	28.0	20.9
Fe_2O_3	%	10.5	10.5
TiO ₂	%	1.0	0.9
CaO	%	4.4	8.1
MgO	%	2.4	4.2
Na ₂ O	%	4.3	1.8
K ₂ O	%	1.5	2.3
SO ₃	%	2.9	6.3
P_2O_5	%	1.2	1.1

Due to the extremely low ash content of sample R1 a good CRI value of 24.1% is calculated based on the coal properties (Figure 3). The CRI value of 25.1% measured at the coke from the retort proves a very acceptable accordance with the calculated value. The different influences of the different coal characteristics – also displayed in Figure 3 – show improving effects of Fe₂O₃, CaO and the reflectance analysis results; slightly deteriorating effects are caused by K_2O , P_2O_5 and the G value.



Figure 3. Influences of different coal characteristics on CRI of coal R1.



Figure 4. Influences of different coal characteristics on CSR of coal R1.

Figure 5 shows the influences of different coal characteristics on the calculation of the CRI value for the coal sample R2 with substantially higher ash content. The considerably high CaO content together with the normally high ash content causes a significant deterioration in CRI. The Fe_2O_3 content of the ash did not change compared with the sample R1, but due to the ash content of sample R2 of 8.42% (mf) this ash component now leads to a quality decreasing effect with respect to CRI. The effects of K_2O and P_2O_5 are more or less negligible. The only quality improving effect is given by the reflectance analysis results.

The zero line in Figure 3 corresponds with a CRI value of 27.9%. This value results from the statistical evaluation and represents the mean value of all the assessed test results. Positive as well as negative deviations of the individual characteristics finally sum up to the calculated value of 24.1% in case of the sample R1.

Figure 4 contains the relevant effects of the coal characteristics on the CSR value of coal sample R1. The zero line corresponds with a CSR value of 57.8%. Besides from the quality improving effects described for CRI two further coal properties contribute to an increasing CSR value: the relatively high bulk density of 880 kg/m³ and the

vitrinite content. Also in this case calculated (71.1 %) and measured value (71.3 %) are in excellent accordance.

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Figure 5. Influences of different coal characteristics on CRI of coal R2.

The accordance between calculated and measured value is much worse than for sample R1. The calculation model seems to put too much emphasis on the catalytic influence of CaO. Thus the calculated CRI value of 44.2% is by 8% (absolutely) higher than the measured CRI of 36.2%.



Figure 6. Influences of different coal characteristics on CSR of coal R2.

The accordance of calculation and measurement is much better for the CSR value of coal sample R2 as shown in Figure 6. Also in this case the measured value of 51.8% is better than the calculated CSR of 48%; but due to the fact that the estimation of coke quality characteristics of samples with such extreme properties by prediction models is very difficult, the difference between calculation and measurement of 3.8% (absolutely) can be considered as satisfactory. The coke quality decreasing effect of CaO is obviously reflected correctly. No other coal property can compensate for this significant negative influence. CRI and CSR are predominantly affected by the mineral matter of the coal^[5]. A prediction method with the aim to include coals of world-wide origin has to make concessions to the accuracy of the estimation. Despite

from that the comparison of calculated and measured values of CRI in Figure 7 and of CSR in Figure 8 for numerous individual coals as well as blends proves a satisfactory accordance between prediction and measurement.

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Figure 7. Comparison of measured and calculated CRI values.



Figure 8. Comparison of measured and calculated CSR values.

The developed prediction method can be applied as useful means to assist in blend optimization.

3 COAL BLEND OPTIMIZATION

Semi-industrial carbonization tests as well as pilot scale tests in the 10-kg-retort are not only practiced to confirm the predicted coke properties. Of course further information are attained concerning the carbonization behavior – especially the swelling pressure – and the conventional drum indices according to ISO 556 (Micum or Irsid values). Furthermore the combined information resulting from calculation, semi-industrial scale and pilot scale carbonization can deliver supplementary valuable conclusions about the carbonization behavior of a blend, as it is described by the following example. The so-called Elk Valley Blend (EVB) is often a major component in typical coking coal blends. EVB consists of a mixture of Elkview and Fording and shows a low volatile matter content of around 22% (mf). The dilatation value of EVB is already negative. Adding pet coke leads very quickly to the situation that only contraction can be monitored in the dilatometer test. Table 4 summaries the coal properties of an EVB sample investigated at DMT. Also the properties of this EVB sample to which 10 % of pet coke had been added are displayed.

ISSN 2176-3135

		Elk Valley Blend	EVB + 10 % PC
Ash (mf)	%	9,5	8,8
Volatile matter content (mf)	%	22,2	21,0
Volatile matter content (maf)	%	24,5	23,1
Free Swelling Index		5,5	4,0
Dilatometer-Test			
Softening point	°C	412	404
Resolidification point	°C	476	464
Plastic range	К	64	60
Contraction	%	24	28
Dilatation	%	-1	Only contraction
G value		0,927	0,000
Max. fluidity (Gieseler-Test)	ddpm	16	7
Mean reflectance R _m	%	1,14	Not determined
Vitrinite content	Vol%	58	Not determined
Bulk density, wet	kg/m³	850	850
Coal moisture	%	8,1	8,0

Table 4. Properties of the Elk Valley Blend without and with addition of pet coke

The coking capacity characterized by the G value shows a result of 0,927 for EVB. Already this value is below the normal range required for typical coking coal blends (0,95 to 1,10). In accordance with the description of the coking capacity by the G value also the determination of the maximum fluidity (Gieseler test) proves a very low value of only 16 ddpm.

The addition of 10% pet coke finally leads to a G value of 0,000 (i.e. only contraction). The maximum fluidity decreases to 7 ddpm proving extremely moderate caking properties. Only the determination of the free swelling index (FSI) shows still existing caking properties. The FSI of EVB is 5,5 and after the addition of 10% pet coke it decreases to 4,0. These caking properties are sufficient to produce a lumpy coke though the dilatometer and the Gieseler test indicate nearly no caking properties. Table 5 contains the ash composition of EVB. Unfavorably high values are not conspicuous. The relatively low value for the Fe2O3 content should be of positive influence on CRI and CSR. The CaO content is in the normal range.

Table 5. Ash composition of the Elk Valley Blend

		Elk Valley Blend
SiO ₂	%	58,0
Al ₂ O ₃	%	28,1
Fe ₂ O ₃	%	3,6
TiO ₂	%	1,7
CaO	%	2,9
MgO	%	0,7
Na ₂ O	%	0,21
K ₂ O	%	0,74
SO ₃	%	1,4
P ₂ O ₅	%	1,3

Figure 9 displays the influences of the different coal properties on the CSR value of EVB. The calculated CSR value and the CSR value measured after the semiindustrial scale trial are included.



Figure 9. Influences of the different coal characteristics on the CSR value of the Elk Valley Blend.

The predominantly positive influences are clearly visible. The slightly negative influence of the CaO content is negligible. The extreme quality reducing effect of the moderate G value is striking. Obviously this can be compensated partly by other influences during carbonization. Thus the coke from the semi-industrial carbonization test resulted in a measured CSR value of 64,7% (4% absolutely higher than predicted).

CRI and CSR values predicted based on EVB properties are compared in Figure 10 with values measured at coke samples from semi-industrial and pilot scale carbonization trials. The calculated values only emphasize on the pure EVB since the G value of 0,000 after pet coke addition does not permit any reliable calculation of CRI or CSR.



Figure 10. Comparison of calculated and measured CRI and CSR values for the Elk Valley Blend (EVB) without and with pet coke addition.

The CRI values of the cokes produced in the carbonization test facilities are by 5 to 7 percentage points better than the calculated value of 30,4%. The differences between the CRI values of the semi-industrial scale test and the pilot scale carbonization are negligible and also the addition of 10% of pet coke to EVB moves the CRI level of the semi-industrial scale coke only very slightly from 25,0 to 23,3% while the CRI values of the retort coke do not change at all.

As already mentioned above the CSR value of the semi-industrial scale coke produced from 100% EVB (64,7%) is by 4 percentage points better than the calculated value (60,7%). But the addition of pet coke to EVB leads anyhow to a decreased CSR value of 61,7% though the CRI value had been improved slightly as described above. Under the conditions of the semi-industrial scale carbonization test oven the decreasing coking capacity of the charged coal has obviously already a negative influence on the formation of a mechanically stable coke matrix.

The CSR values monitored at the retort coke samples prove an absolutely constant level. Under the carbonization conditions in the 10-kg-retort which are characterized by a higher carbonization rate and a maximum of cracking reactions of the raw gas in the incandescent coke the decisively reduced caking properties have obviously no negative influence on the formation of a mechanically stable coke matrix.

The described different behavior of coals in the semi-industrial scale oven and in the 10-kg-retort as well as the indicated differences between calculation and carbonization tests only occur in case of very lean coal charges with extremely moderate caking properties. The most extreme differences ever monitored at such a coal between semi-industrial oven coke and retort coke amounted to 20 percentage points in CSR. The calculated values always were much closer to the CSR level of the semi-industrial oven coke. For individual coals and blends with normal caking properties the differences are nearly negligible.

Nevertheless, important conclusions for coke plant operators can be derived from the correlations described. These conclusions are valid for each lean blend with very moderate caking properties (e.g. blends with high quantity of EVB or any similar coal).

The local carbonization conditions are decisive for the formation of a mechanically stable coke matrix from such blend. As well known the carbonization conditions in the

industrial scale oven are by far not constant, dependent on the location in the oven chamber they may vary significantly^[6]. E.g. the carbonization rate changes with the distance from the heating wall. Furthermore even the coal properties are influenced since hydrocarbons of the raw gas are condensed in the cold centre of the chamber and thus affect coking properties. It has been proved that both phenomena lead to the effect that the coke quality in the centre of the chamber is negatively influenced^[3]. The quality difference between centre coke and wall coke is the more significant the leaner the coal blend that has been charged.

ISSN 2176-3135

But there are even more influences to be considered. Also the bulk density of the oven charge varies dependent on the distance from the oven sole^[7]. Cracking reactions of the raw gas in the incandescent coke are favored in wide and high chambers, due to the prolonged residence time of the raw gas in contact with the hot coke. Furthermore local differences can also be traced back to extreme quantities of condensed water vapors. Such accumulation areas of water are randomly distributed in the oven charge and lead to a delayed carbonization of this part of the charge^[8].

The description shows quite numerous phenomena which may cause local differences in carbonization conditions in an industrial scale coke oven. The charging of a very lean blend at a coke plant may obviously lead to a wider scattering of the quality characteristics of the produced coke – especially with respect to CRI and CSR – though operating the plant with a constant blend composition, but with more economical benefit.

4 COKE OVEN SAFETY

Coke oven operators use lances for the measurement of the internal gas pressure. The perforated tube on the lance head passes through the coke oven door or the charging holes. The generated gas during the carbonization push against the gas column in the pipe and a pressure transmitter registers the pressure change. Figure 11 shows the lance head of the internal gas pressure measurement system that is used by DMT.



Figure 11. Internal gas pressure lance at DMT.

Coke oven operators try to confirm their measured maximum internal gas pressure when a higher pressure value in the oven is expected or exists. A test with the coal blend outside of the industrial coke oven can increase the reliability for the optimization of the coal blend in relation to coke oven safety. The well known smallscale coking test retort is a possible test facility. DMT changes two main points in the test procedure in comparison to the standard procedure for the coke quality evaluation (see chapter 2.2). When coke oven safety is concerned follow changes are to be considered:

ISSN 2176-3135

- Adjustment of the operating condition in small-scale coking test retort to the conditions that are present at the internal gas pressure test point in the industrial coke oven
- DMT takes another approach for the interpretation of the obtained pressure curve during carbonization in the small-scale coking test retort to overcome this problem.

DMT usually adjusts the average bulk density of the industrial coke oven in the smallscale coking test retort to get valuable information about the coke quality. The measurement of the internal gas pressure generally occurs in deeper layers of the coal bed. The adjustment of the average bulk density of the industrial coke oven leads to inaccurate pressure values due to the high influence of the bulk density on the internal gas pressure. DMT conducted some measurements of the bulk density distribution in industrial coke ovens. These data and the Janssen equation for silo design allow the calculation of the existing bulk density at the internal gas pressure testing point in the industrial coke oven. Figure 12, for instance, illustrates the bulk density distribution at a German coking plant with 6 m high coke ovens. Figure 12 displays that the existing bulk density at the test point is more than 100 kg/m³ higher than the average bulk density.



The second point is the interpretation of the obtained pressure curve. Figure 13 gives an overview of the difference between the pressure diagram that were obtained in the semi-technical or industrial coke oven and the small-scale coking test retort with the same coal blend.



Figure 13. Comparison of the pressure progression in semi-technical and small scale coke oven.

The left diagram reproduces the typical internal gas pressure and center plan temperature progression in the semi-technical or industrial coke oven during the carbonization. The pressure reaches a maximum at a center charge temperature of 450 – 500°C. This corresponds to the closing of the plastic layers in the central plane of the oven. The interrupted red line on the right side of Figure 13 indicates a confused progression of internal gas pressure of the small-scale coking test retort. A theoretical approach about the behavior of the plastic layer during the carbonization can clarify this progression.

In contrast to the semi-technical and industrial scale coke ovens the plastic layer in the small-scale coking test retort quickly generates a closed envelope which is similar to an ellipsoid. The ellipsoid compresses the enclosed water vapors which are forced against the plastic layer. The first pressure peak in Figure 13 right hand side shows the counteracting of the plastic layer. The plastic layer resists the water pressure for some minutes before get first fissures and the vapors escape in the semi-coke. After the recombination of the plastic layer. In the meantime the volume of the ellipsoid declines. The pressure peaks increase faster and reach higher values. The high pressure generates fissures in the plastic layer and the pressure immediately drops down. The plastic layer recombines and the pressure rises again. This act takes so long as the centre temperature rise up to $350 - 500^{\circ}$ C and the ellipsoid shrink to a small ball that generates the last pressure peak.

This behavior during the carbonization can be interpreted as the resistance of the plastic layer against the pass of the evolved gases. Nomura et al. shows in their investigation the correlation between highest pressure force to go through the plastic layer and the measured maximum internal gas pressure in a semi-technical movable coke oven^[9]. The new interpretation of the pressure progression during the carbonization in the small-scale coking test retort and a test program of 50 tests in the small-scale and industrial scale oven allow DMT to generate a prediction model. The prediction model allows with the extracted pressure data to estimate the maximum internal gas pressure in the industrial coke oven. Figure 14 displays the calculated maximum internal gas pressure and the measured values at the industrial coke oven.



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Figure 14. Comparison of measured and calculated max. int. gas pressure.

The accuracy of the model is in the range of \pm 30 mbar. The results of the new approach gives the small-scale coking test retort the possibility to be a fast and cost effective test method to consolidate the measured internal gas pressure in the industrial coke oven and to increase the reliability of the coke oven operator for the optimization of the coal blend in terms of coke oven safety.

5 CONCLUSIONS

DMT with their experience and evolution of the above mentioned research work developed a procedure for the determination of an optimal coal blend in terms of coke quality, coke oven safety and economics. DMT applies the following steps:

- The first approach of blend optimization is the complete analysis of single coals followed by calculations with the DMT prediction model concerning quality requirements and economic aspects
- Confirmation of the calculated results with some (6-10 tests) in the pilot scale carbonization oven (10-kg-retort)
- Comprehensive assessment of the carbonization behavior and coke quality through selected semi-industrial carbonization tests. Selection of tests determined by the initial two steps

The aforementioned procedure and the reliable determination of coking behavior (Internal gas pressure, wall load and shrinkage) and coke quality will be essential information for the coke plant operator to achieve the required coke quality and production targets.

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