Crude oil sources and cut point as keys for the prediction of the mechanical performance of bitumen

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ABSTRACT: The research objective was to investigate the chemical composition and rheological properties in order to identify potential key parameters for the prediction of bitumen performance parameters. Together with Austria's largest bitumen producer OMV two extensive experimental programs were carried out. The programs involved empirical, rheological, chemical, physical and micro-structural analysis with a vast number of bitumen samples from different crude oil sources distilled at different cut points. To assess the complex chemical composition of bitumen elemental analysis (EA), metal content determination (metal. C), gel permeation chromatography (GPC) and the iatroscan method were used. In order to link the obtained chemical composition to rheological properties, dynamic shear rheometer (DSR), bending beam rheometer (BBR) and rotational viscosimeter tests (RV) were carried out providing material properties in aged and unaged condition in a temperature range between -24°C to 180 °C. Based on a statistical factor analysis it was possible to predict conventional parameters (PEN, ERK, FRAAS etc.) and rheological parameters (Gstar 64°C to Gstar -20°C, Superpave High PG and low PG etc.) for both bitumen derived from single crude oil sources and their blends by means of fast and straightforward experiments. The findings of the research support the evaluation of the derived bitumen quality prior to buying a crude oil with substantial reduced testing effort.

KEY WORDS: Cut, point, bitumen, performance, optimization

1 INTRODUCTION

From a general perspective concerning bitumen and asphalt concrete pavements, there are several important steps towards a sustainable use of natural resources and lasting and safe road infrastructures (Figure 1). This paper specifically addresses the refining process (Step 2) and assessment of the bitumen performance (Step 3) in this life cycle approach.

The conventional and rheological parameters of bitumen are well defined for the European standards. In order to meet these standards or to produce bitumen with preset parameters from different blends of crude oils, usually extensive testing prior to the refining process is necessary. Furthermore, it is quite difficult to obtain the optimal mix proportion and cut point for the distillation in such a process. This paper presents a supportive tool to meet these requirements based on the extensive experimental programs and the statistical analysis of the derived data.

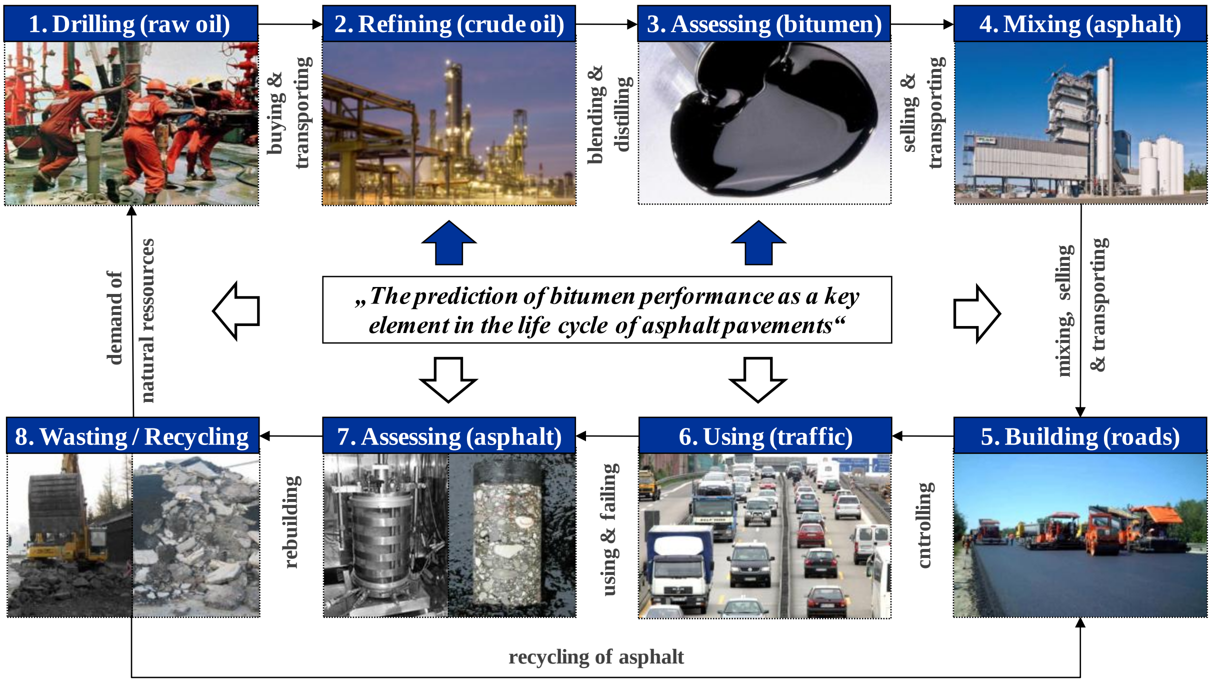


Figure 1: A life cycle approach - from crude oil to asphalt and back.

2 EXPERIMENTAL PROGRAM AND FRAMEWORK

2.1 Identification of key parameters

Bitumen is the remaining part of crude oil distillation and has a complex chemical mixture of organic molecules that show a wide variation from non-polar saturated hydrocarbons to highly polar and condensed aromatic systems. Different crude oils have different chemical and physical characteristics and therefore show a different mechanical performance at the same cut point (Shell-Bitumen-UK, 1990).

In the continuous vacuum distillation the lighter fractions (Petroleum, Gasoline, Kerosene and Diesel) are drawn to the top of the tower while the residue remains at the bottom. The higher the cut point temperature gets, the lighter the fractions become when they are distilled and the heavier and less penetrable the residue gets. The penetration grade (PEN) at 25°C for instance is a conventional parameter and one of the key features according to the EUROCODE EN 12591 in order to distinguish between different types of bitumen and their possible field of use.

During the mixing process in the asphalt plant and the building process the bitumen is heated up again which introduces chemical changes and further hardening due to oxidation and loss of volatile components. This process is called short-term ageing (state B). After 5 to 10 years of service life tests show a further hardening called long-term ageing (state C). This aging process of bitumen is one of the key parameters in the cracking mechanism of asphalt pavements and can be simulated with performance-based testing methods (LU & ISACSON 1998, 2002). According to SUPERPAVE (2003), short-term ageing effects resulting from mixing, transportation and paving can be simulated in the rolling thin film oven (RTFOT). Long-term ageing due to oxidation and weathering conditions representing a state after 5 to 10 years of service life can be obtained in the pressure-ageing vessel (PAV) tests.

2.2 Experimental programs

For the prediction of the mechanical performance parameters of bitumen, an extensive database is needed for statistical analyses. In this analysis already known correlations have to be verified in order to obtain reliable master functions. Based on these functions it was possible to obtain reliable prognosis results even with small samples of data. The presented findings in this paper are based on the results of the experimental programs of STANGL (2008) at the Christian Doppler – Labs at the Technical University of Vienna (Table 1) and OMV (2008) at the Bitumen Labs of OMV (Table 2).

The link between chemical and physical characteristics and its resulting conventional and rheological properties were investigated from STANGL (2008) based on 42 bitumen samples in aged (B, C) and unaged (A) condition from four different crude oil sources, three cut points and two control samples of unknown provenience.

Table 1: Experimental program at the Christian Doppler Laboratories (STANGL 2008)*.

Source (Cutpoint)	Conventional tests									Rheological characterization											
Method	*Pen*			*SP*			*Fraass*			*RV*			*DSR*			*ZSV*			*BBR*		
Aging Stage	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*
Control #1 50/70	x		x	x		x	x			x	x	x	x		x	x		x	x		x
Control #2 PmB 45/80-65	x		x	x		x	x			x	x	x	x		x	x		x	x		x
Crude oil #1 (480)	x			x			x			x	x	x	x		x	x					x
Crude oil #1 (515)	x		x	x		x	x			x	x	x	x		x	x					x
Crude oil #1 (555)	x			x			x			x	x	x	x		x	x					x
Crude oil #2 (525)	x			x			x			x	x	x	x		x	x					x
Crude oil #2 (545)	x			x			x			x	x	x	x		x	x					x
Crude oil #2 (560)	x		x	x		x	x			x	x	x	x		x	x					x
Crude oil #3 (510)	x			x			x			x	x	x	x		x	x					x
Crude oil #3 (540)	x			x			x			x	x	x	x		x	x					x
Crude oil #3 (560)	x		x	x		x	x			x	x	x	x		x	x					x
Crude oil #4 (410)	x			x			x			x	x	x	x		x	x					x
Crude oil #4 (485)	x		x	x		x	x			x	x	x	x		x	x					x
Crude oil #4 (525)	x			x			x			x	x	x	x		x	x					x

Source (Cutpoint)	Chemical, physical and microstructural analysis																				
Method	*Density*			*Gain*			*EA*			*Metal-C.*			*GPC*			*Iatroscan*			*MDSC*		
Aging Stage	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*
Control #1 50/70	x						x	x	x				x	x	x	x	x	x	x	x	x
Control #2 PmB 45/80-65							x	x	x				x	x	x	x	x	x	x	x	x
Crude oil #1 (480)	x						x						x			x	x	x	x		
Crude oil #1 (515)	x						x		x	x		x	x		x	x	x	x	x		
Crude oil #1 (555)	x						x						x			x	x	x	x		
Crude oil #2 (525)	x						x						x			x	x	x	x		
Crude oil #2 (545)	x						x						x			x	x	x	x		
Crude oil #2 (560)	x						x		x	x		x	x		x	x	x	x	x		
Crude oil #3 (510)	x						x						x			x	x	x	x		
Crude oil #3 (540)	x						x						x			x	x	x	x		
Crude oil #3 (560)	x						x		x	x		x	x		x	x	x	x	x		
Crude oil #4 (410)	x						x						x			x	x	x	x		
Crude oil #4 (485)	x						x		x	x		x	x		x	x	x	x	x		
Crude oil #4 (525)	x						x						x			x	x	x	x		

A… *original unaged stage* ***B…*** *RTFOT aged stage* ***C…*** *RTFOT + PAV aged stage*

In addition, the OMV conducted a research on five crude oil sources and their possible qualification for the distillation/processing/production. Furthermore, one polymer modified control sample and two defined blends of crude oils were analyzed. The main emphasis of this experimental program was the establishment of reliable correlations between cut point and conventional parameters of bitumen by means of bivariate exponential regression models.

Table 2: Experimental program at the Bitumen Labs of OMV (OMV 2008)*

Source (Cutpoint)	Conventional tests									Rheological characterization											
Method	*Pen*			*SP*			*Fraass*			*RV*			*DSR*			*ZSV*			*BBR*		
Aging Stage	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*
Control #1 PmB 60/90	x	x		x	x		x	x		x											
Blend #1 (410)	x			x						x											
Blend #1 (500)	x			x						x											
Blend #1 (540)	x			x						x											
Blend #1 (560)	x			x						x											
Blend #2 (520)	x			x						x											
Blend #2 (540)	x			x						x											
Crude oil #1 (410)	x	x		x	x		x	x		x											
Crude oil #1 (500)	x	x		x	x		x	x		x											
Crude oil #1 (560)	x	x		x	x		x	x		x											
Crude oil #5 (410)	x	x		x	x		x	x		x											
Crude oil #5 (500)	x	x		x	x		x	x		x											
Crude oil #5 (560)	x	x		x	x		x	x		x											
Crude oil #6 (410)	x	x		x	x		x	x		x											
Crude oil #6 (500)	x	x		x	x		x	x		x											
Crude oil #6 (560)	x	x		x	x		x	x		x											
Crude oil #7 (410)	x	x		x	x		x	x		x											
Crude oil #7 (500)	x	x		x	x		x	x		x											
Crude oil #7 (560)	x	x		x	x		x	x		x											
Crude oil #8 (410)	x	x		x	x		x	x		x											
Crude oil #8 (485)	x	x		x	x		x	x		x											
Crude oil #8 (506)	x	x		x	x		x	x		x											
Crude oil #8 (513)	x	x		x	x		x	x		x											
Crude oil #8 (525)	x	x		x	x		x	x		x											

Source (Cutpoint)	Chemical, physical and microstructural analysis																				
Method	*Density*			*Gain*			*EA*			*Metal-C.*			*GPC*			*Iatroscan*			*MDSC*		
Aging Stage	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*
Control #1 PmB 60/90	x			x												x					
Blend #1 (410)	x			x																	
Blend #1 (500)	x			x																	
Blend #1 (540)	x			x																	
Blend #1 (560)	x			x																	
Blend #2 (520)	x			x																	
Blend #2 (540)	x			x																	
Crude oil #1 (410)	x	x		x												x					
Crude oil #1 (500)	x	x		x												x					
Crude oil #1 (560)	x	x		x												x					
Crude oil #5 (410)	x	x		x												x					
Crude oil #5 (500)	x	x		x												x					
Crude oil #5 (560)	x	x		x												x					
Crude oil #6 (410)	x	x		x												x					
Crude oil #6 (500)	x	x		x												x					
Crude oil #6 (560)	x	x		x												x					
Crude oil #7 (410)	x	x		x												x					
Crude oil #7 (500)	x	x		x												x					
Crude oil #7 (560)	x	x		x												x					
Crude oil #8 (410)	x	x		x												x					
Crude oil #8 (485)	x	x		x												x					
Crude oil #8 (506)	x	x		x												x					
Crude oil #8 (513)	x	x		x												x					
Crude oil #8 (525)	x	x		x												x					

A… *original unaged stage* ***B…*** *RTFOT aged stage* ***C…*** *RTFOT + PAV aged stage*

2.3 Prediction framework and program

Based on a bivariate and multivariate regression analysis the key factors explaining the relationships between chemical/physical and performance parameters of bitumen in a temperature range from -24°C to 180 °C could be identified. In a factorial analysis STANGL (2008) was able to identify two underlying factors that explain the pattern of correlation within the observed bitumen parameters.

As a result, all performance parameters and the low temperature glass transition Tg,low loaded highly on one factor, whereas the remaining chemical and physical bitumen characteristics indicate correlations of high temperature properties with density and average molecular weight in a second factor. By means of this factor analysis the correlation between chemical/physical characteristics and performance parameters in the aged and unaged stage could be established. Furthermore, with the derived regression equations it is possible to estimate the bitumen performance for high and low temperature regimes with fast and straightforward experiments like gel permeation chromatography (GPC), elemental analysis or density.

The experimental program and analysis of the data at the Bitumen Labs of OMV (OMV 2008) showed strong relationships between the conventional bitumen parameters and the bitumen density with the cut point. In many cases exponential bivariate regressions resulted in a good fit with an R squared of 0.9 or higher. For some of the tested crude oil sources, the high paraffin content was a limiting factor for an unblended use in the refining process. Therefore a good prediction model should also be able to forecast the performance of any blend from already analyzed crude oil sources in order to obtain the optimal mix proportions.

The presented framework in Figure 2 combines these approaches in order to allow a prediction of bitumen or blend performance for both conventional and performance-based properties prior to distillation mainly based on crude oil source, mix proportion and cut point. In the current version a database and the performance prediction for crude oil and their blends are realized and will be dynamically updated with each new sample of data.

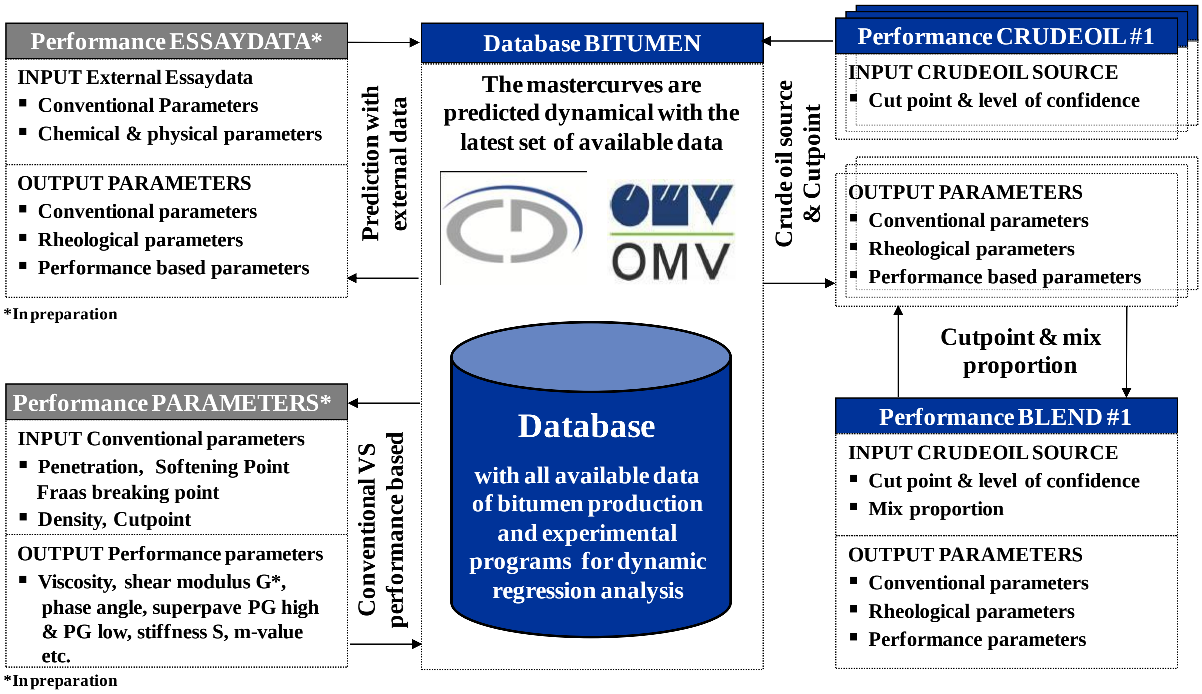


Figure 2: Key parameters and framework for the performance prediction of bitumen.

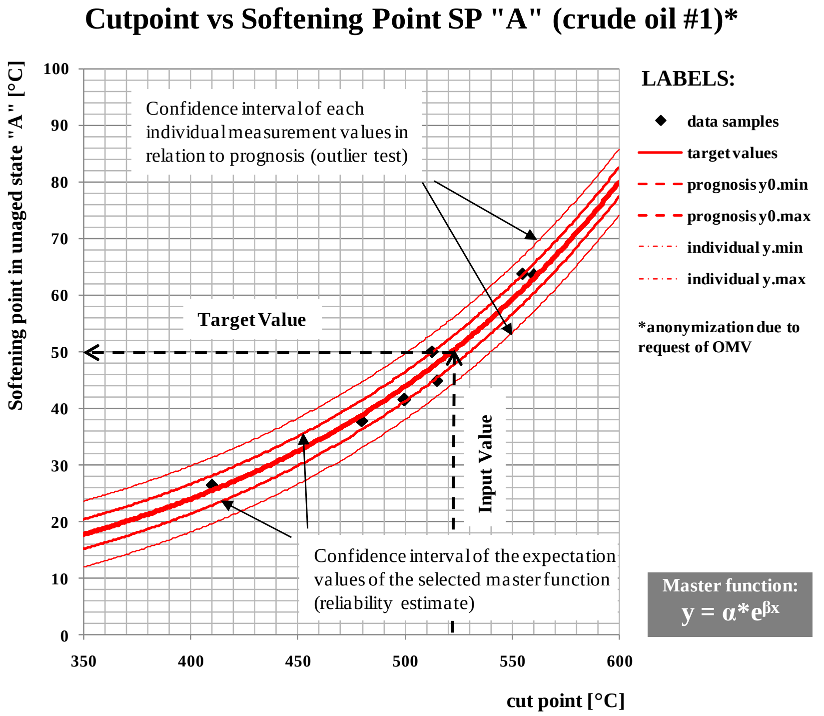
3 SELECTED RESULTS OF THE PREDICTION TOOL

3.1 Master curves

The prediction in the MS EXCEL 2003 based tool shows the regression parameters and the target value for several statistical functions (Table 3). With the selection of a specific function the target value can be predicted for a range of cut points from 350 °C to 600 °C (crude oil #1). In addition, the confidence interval is given for the selected master function (inner interval) and the individual data (outer interval) from conventional and rheological testing (Figure 3). Once the best fitting master curve is set as default for each target value, the prediction is automatically updated with each new data sample from bitumen tests for each crude oil source. Thus the prediction becomes more solid over time. Furthermore, the prediction tool helps to identify the cut point for the distillation with the highest likelihood of obtaining the desired performance. Other influence factors are already included due to the restriction to one crude oil source at a time of the regression.

Table 3: Softening point (SP) prediction with the available functions in the software tool*

	Parameters of the currently available bivariate master functions												
Master functions	y=α+βx	y=a+ß/x	y=αβ^x	y=αx^β	y=αe^βx	y=αe^β/x	y=α/(β+x)	y=α+βln(x)	y=α+β1x+β2x^2	y=α+β1x+... +β3x^3	y=α+βln(ln(x))	y=α+β1sin(β2x)	y=α+β1/(1+e^(β2+β3x))
Input value: cut point x	520	520	520	520	520	520	520	520	520	520	520	520	520
Input value: level of confidence	0,95	0,95	0,95	0,95	0,95	0,95	0,95	0,95	0,95	0,95	0,95	0,95	0,95
Data samples	8	8	8	8	8	8	8	8	8	8	8	8	8
Parameter α	-82,759226	160,444621	0,77822249	-14,056992	0,77822249	6,51902952	0,09838139	-709,7454497	196,3093504	529,5319831	-1320,991469	58,91766423	23,76884835
Parameter β1	0,25651527	-56907,408	0,00600563	2,87248486	0,00600563	-1355,8371	-0,0001493	121,6202407	-0,903995428	-2,977275543	748,3484236	32,28524229	62,17673237
Parameter β2	0	0	0	0	0	0	0	0	0,001194527	0,005459845	0	0,011524238	12,9263164
Parameter β3	0	0	0	0	0	0	0	0	0	-2,90357E-06	0	0	-0,024211007
Regression coefficient R^2	0,923	0,863	0,975	0,963	0,975	0,945	0,984	0,895	0,980	0,981	0,890	0,980	0,981
R^2adjusted	0,910	0,841	0,971	0,957	0,971	0,936	0,981	0,878	0,977	0,977	0,872	0,977	0,978
Target Value	50,63	51,01	49,46	49,75	49,46	49,98	48,21	50,85	49,23	49,43	50,88	49,67	49,67
Lower bound prognosis y_{0min}	47,21	46,40	47,53	47,36	47,62	47,06	46,63	46,83	47,35	47,33	46,99	47,78	47,60
Upper bound prognosis y_{0max}	54,05	55,62	51,39	52,14	51,30	52,91	49,79	54,86	51,11	51,52	54,76	51,55	51,75
Lower bound individual data y_{min}	40,81	37,92	43,90	42,92	43,93	41,68	43,67	39,38	43,82	43,40	39,22	44,25	43,71
Upper bound individual data y_{max}	60,45	64,10	55,02	56,58	54,98	58,29	52,75	62,31	54,64	55,45	62,53	55,08	55,64
Selected master function (1)	○	○	○	○	◉	○	○	○	○	○	○	○	○

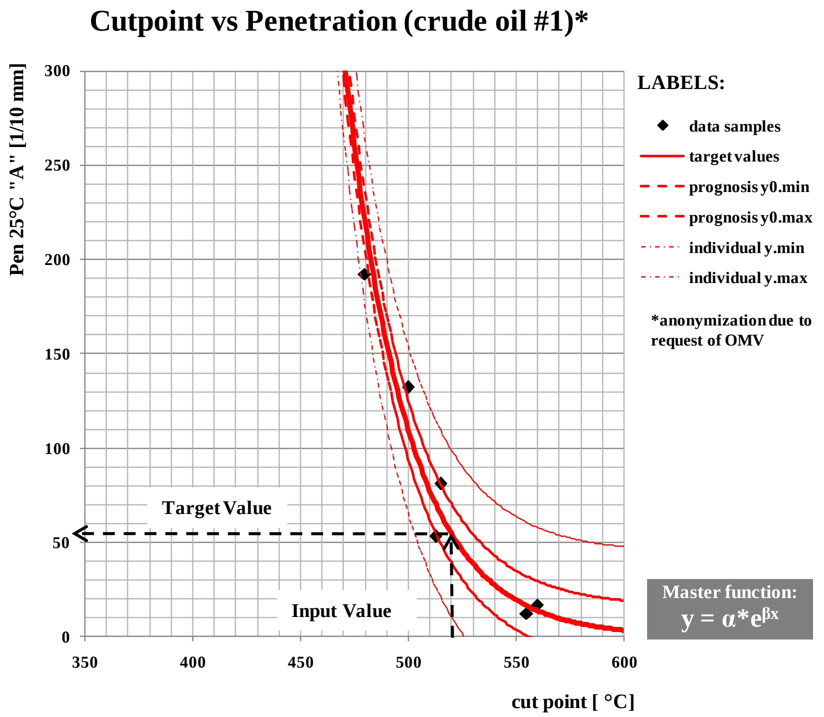


Master functions	y=αe^βx
Input value: cut point x	520
Input value: level of confidence	0,95
Data samples	8
Parameter α	0,778222491
Parameter β1	0,006005625
Parameter β2	0
Parameter β3	0
Regression coefficient R^2	0,98
R^2adjusted	0,97
Target Value	49,5
Lower bound prognosis y_{0min}	47,6
Upper bound prognosis y_{0max}	51,3
Lower bound individual data y_{min}	43,9
Upper bound individual data y_{max}	55,0

Figure 3: Softening point (SP) prediction for the selected master functions with software tool.

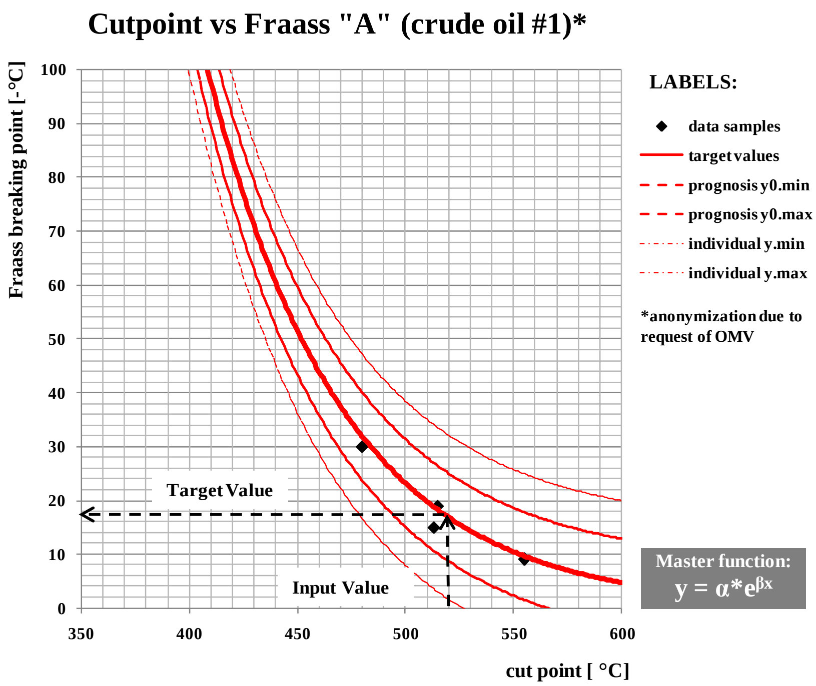
3.2 Prediction of conventional bitumen parameters

The trend of hardening of the resins with an increasing cut point during the refining process results in an increasing Softening Point SP (Figure 3), decreasing Penetration Grade PG (Figure 4) and a lowered Fraass breaking point (Figure 5) for all tested crude oil sources. This consistent trend is also of high importance for the calculation of rheological parameters from conventional parameters with the Softening Point as explaining factor.



Master functions	y=αe^βx
Input value: cut point x	520
Input value: level of confidence	0,95
Data samples	7
Parameter α	22,0500
Parameter β1	-0,0347
Parameter β2	0
Parameter β3	0
Regression coefficient R^2	0,95
R^2adjusted	0,94
Target Value	55
Lower bound prognosis y_{0min}	39
Upper bound prognosis y_{0max}	71
Lower bound individual data y_{min}	11
Upper bound individual data y_{max}	99

Figure 4: Penetration (PEN) prediction for the selected master functions.



Master functions	y=αe^βx
Input value: cut point x	520
Input value: level of confidence	0,95
Data samples	5
Parameter α	11,14642
Parameter β1	-0,01601
Parameter β2	0
Parameter β3	0
Regression coefficient R^2	0,82
R^2adjusted	0,76
Target Value	-16,8
Lower bound prognosis y_{0min}	-11,1
Upper bound prognosis y_{0max}	-22,6
Lower bound individual data y_{min}	-2,7
Upper bound individual data y_{max}	-31,0

Figure 5: Fraass breaking point prediction for the selected master functions.

3.3 Prediction of rheological bitumen parameters

For high temperatures the viscosity of bitumen is tested with the rotational viscosimeter (RV) unaged (A) and after ageing in the rolling thin film oven RTFOT (B) and further ageing in the pressure-ageing vessel PAV (C). The results show an increasing viscosity at 135°C in the RV with an increasing cut point for ageing state "A" (Figure 6) and a further hardening due to the ageing process for all samples in state "A", "B" and "C" (Figure 7). The quantification of this hardening at the temperature ranges of the life cycle is of high importance for the resulting service life of asphalt pavements.

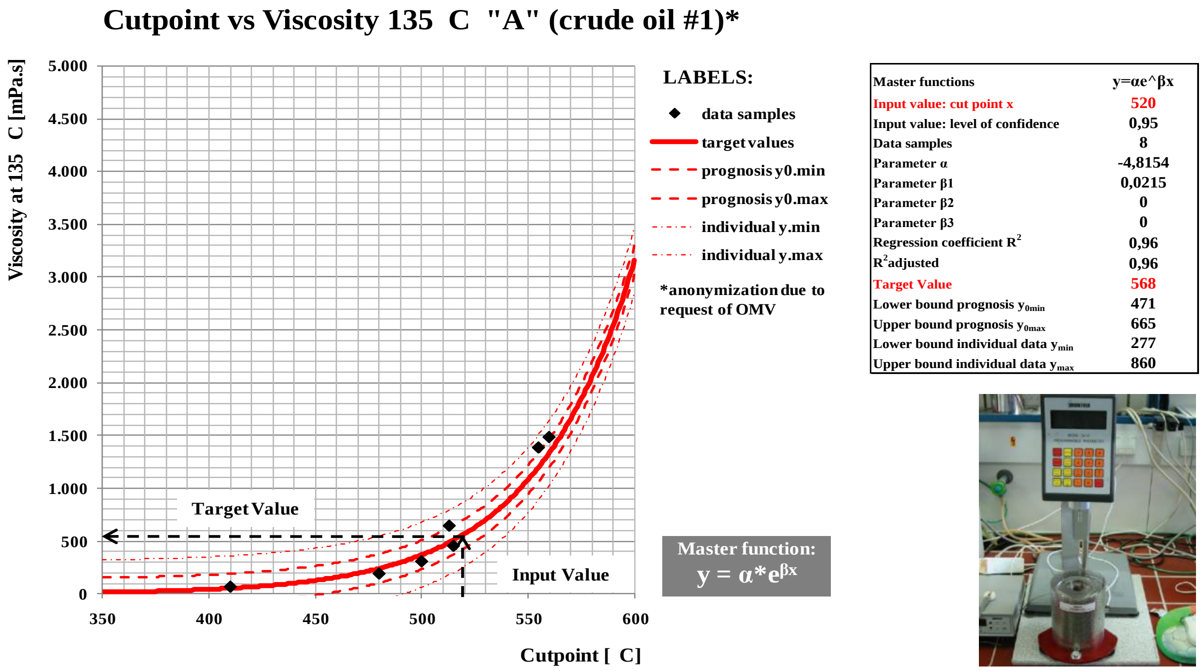


Figure 6: Viscosity prediction for the selected master functions in an unaged state (A).

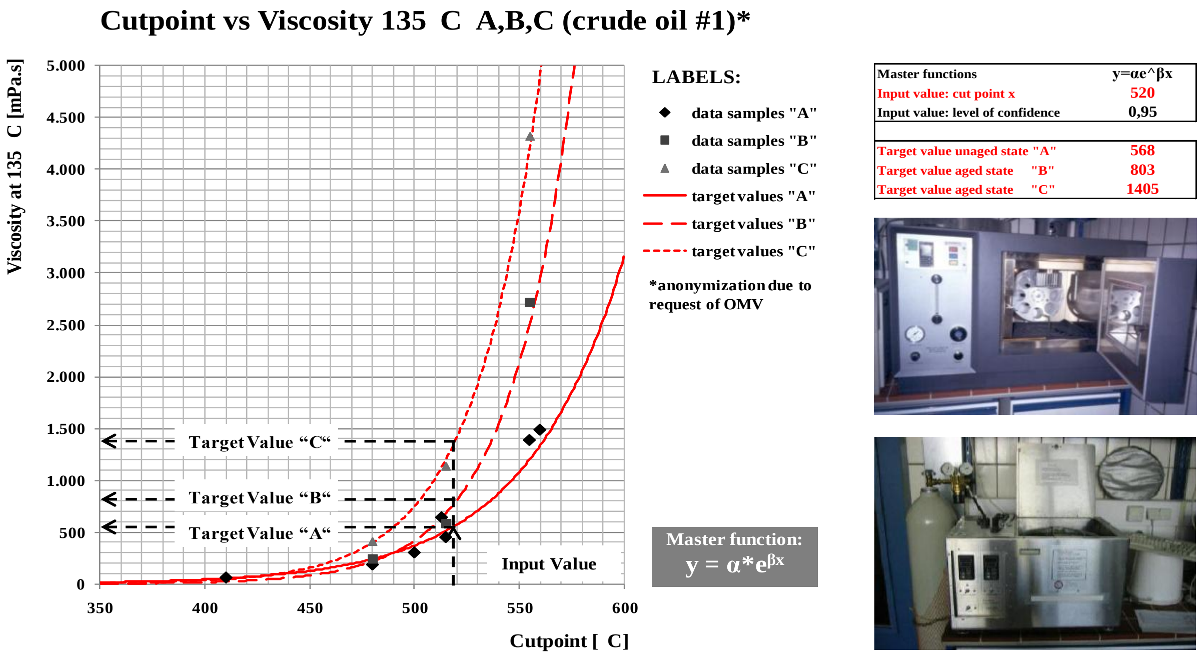
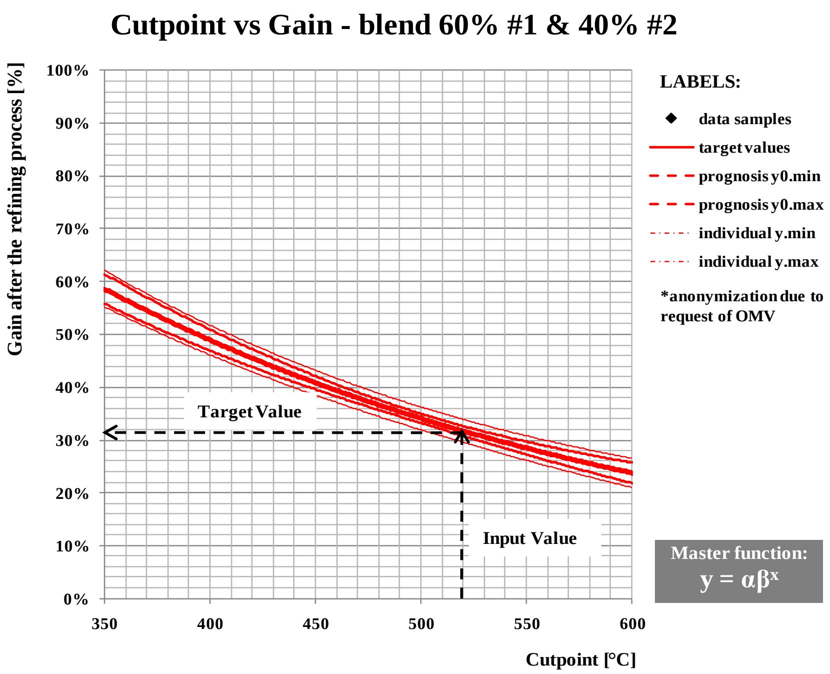


Figure 7: Viscosity prediction in unaged (A) and aged condition (B, C).

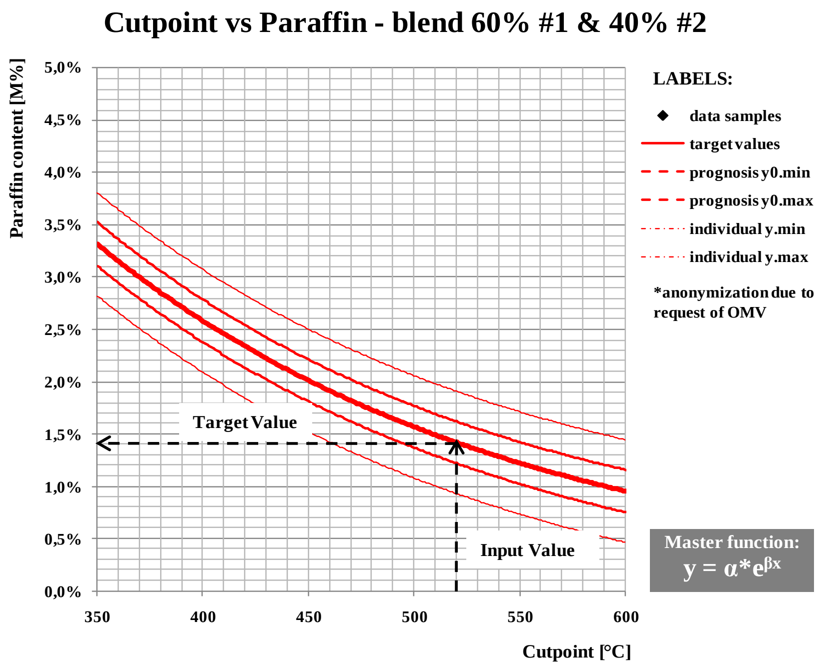
3.4 Prediction of blend parameters

By means of the refinery's long time data base the developed tool also helps to predict the parameters of any blend with different mix proportions through the calculated unblended master curves for each crude oil source at different cut points. If the parameters of bitumen from one crude oil source and a specific blend are known, the influence of the other crude oil source may be identified as well. Furthermore, it is possible to estimate the gain of any blend and its paraffin content allowing the use of otherwise not suited crude oil sources in the refining process (Figure 8, 9).



Master functions	y = αβ^x
Input value: cut point x	520
Input value: level of confidence	0,95
Data samples	5
Parameter α	0,7362
Parameter β1	-0,0036
Parameter β2	0
Parameter β3	0
Regression coefficient R^2	0,99
R^2adjusted	0,99
Target Value	31,7%
Lower bound prognosis y_{0min}	30,8%
Upper bound prognosis y_{0max}	32,7%
Lower bound individual data y_{min}	29,5%
Upper bound individual data y_{max}	33,9%

Figure 8: Gain prediction for the refining process for a blend of 60% #1 & 40% #2.



Master functions	y=αe^βx
Input value: cut point x	520
Input value: level of confidence	0,95
Data samples	5
Parameter α	-1,6532
Parameter β1	-0,0050
Parameter β2	0,0000
Parameter β3	0,0000
Regression coefficient R^2	0,95
R^2adjusted	0,93
Target Value	1,42%
Lower bound prognosis y_{0min}	1,22%
Upper bound prognosis y_{0max}	1,62%
Lower bound individual data y_{min}	0,93%
Upper bound individual data y_{max}	1,91%

Figure 9: Paraffin content prediction for a blend of 60% #1 & 40% #2.

4 CONCLUSIONS AND OUTLOOK

With the development of performance based bitumen and asphalt testing methods during the last decade the conventional bitumen parameters have become less important. Thus for the bitumen industry it is crucial to know which crude oil is bitumen compatible, how their products perform in this new performance based tests, and which possibilities they have to enhance the results of the production process. An additional limitation of the bitumen production is the necessity to blend different crude oil sources in order to achieve a bitumen fraction with the targeted properties without extensive testing for each fraction.

Based on data from extensive research programs at OMV and TU Vienna a prediction tool for conventional parameters (PEN, ERK, FRAAS etc.) and rheological parameters (Viscosity, G*@64°C, G*@-20°C, Superpave High PG & low PG etc.) for both crude oil sources and their blends was developed. Based on the presented prediction framework, bivariate regressions are sufficient due to the separated calculation of the master curves from each crude oil source. Therefore the prediction tool needs only very few input data and helps to reduce the necessary testing effort.

The established master curves can help/support the Quality Management of the refinery to assess bitumen from available crude oil data and supports in the optimization of the blending and refining processes. Further possible uses of the prediction tool on the workflow are summarized in Figure 10. However there are some remaining gaps for some parameters in the database resulting in a somewhat wide confidence interval. An automatic update of the master functions and confidence intervals with each new data sample from bitumen tests for each crude oil source will help to overcome this drawback in the future. Thus the prediction tool will become more and more reliable over time.

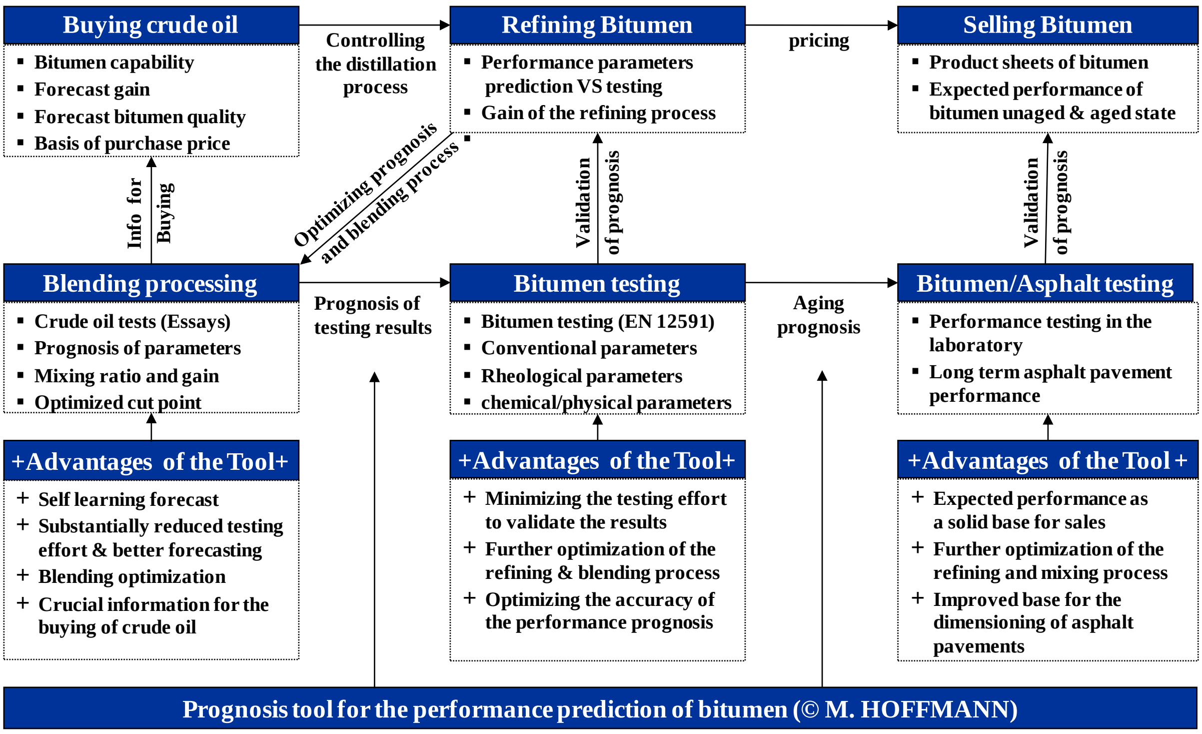


Figure 10: Workflow of the refining process and possible impacts of the prediction tool.

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