Manual for SAXSMorph program (Version 2.2)

B. Ingham

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Industrial Research Ltd., P.O. Box 31310, Lower Hutt, New Zealand

1 Introduction

The program SAXSMorph is a free program for generating a visual representation of three-dimensional structure from SAXS (small-angle x-ray scattering) or SANS (small-angle neutron scattering) data. It is complemented by SAXSFit. These two programs were previously combined in one program called SAXSConvert (see Section 2, 'Development').

The morphology is calculated for a specified box size from extrapolating a specified range of the raw data and using a known porosity value. The user can specify various display options, including setting the scale and resolution, specifying whether to show a 2D slice or 3D representation, to show the solid or pore, to highlight the largest pore. The program also calculates the surface-to-volume ratio and determines if the morphology is bicontinuous.

This program is written in the Java programming language (using Java 2 SDK, version 1.5.0). It is available 'wrapped' in a single .exe file for use on Windows platforms, and as a .jar Java executable for other platforms. It follows the algorithms of a Matlab-based program written by Haiyong Li, which itself was built upon a program written in C-plot and OpenDX by Jonathan Hedstrom. The advantage of using Java is to provide a program which is compatible on several different platforms. The morphology is visualised using POV-Ray, a freeware ray-tracer program which must be downloaded separately (www.povray.org).

Users must be aware of the limitations of the methods used in SAXSMorph for determining the representative morphologies. At present, SAXSMorph can only be applied to two-phase systems that are isotropic and non-periodic. In addition, if the q-range of the data does not capture all relevant length scales the results may hold little meaning (particularly evident in cases where q does not tend to q^{-4} at high q; this may indicate fractal structure or that the data range collected is insufficient).

The user is responsible for understanding the applicability of this approximation to their system, and ensuring that the results are physically meaningful.

2 Development

The SAXS Morphology program is an offshoot of the original SAXS Converter program which was developed to assist in the fitting of SAXS and SANS spectra and generate a visual representation of the three-dimensional structure.

The morphology is calculated from extrapolating the raw data and using a known porosity value (see Section 5 for mathematical details). The morphology file is written as it is calculated and intended to be visualised using POV-Ray.

SAXSConvert parent: Version 1.0 completed October 2005.
Subsequent alterations:

1.1 - 1.7 Alterations to the SAXSFit part of the program. (November 2005 - July 2006)

2.0 Split program to separate fitting and morphology - fitting remains as SAXSConvert, now called SAXSFit, but morphology is now a new program, SAXSMorph. Check if log file already exists; if it does, append to the log file and display a message advising the user that result files will be overwritten unless the output file is changed. (July 2006)

2.1 Add in more user options: select color(s); option for solid, pore, largest pore, 2D or 3D; options for q extrapolation (number of points, to do it or not); fix error handling; show the various plots as they are calculated; calculate statistics on the morphology (continuity); separate the calculation of the Gaussian random field from .pov file generation (create the pore/solid matrix and then manipulate). (December 2008)

2.2 Improve user-friendliness - error catching. Improve plotting. Allow user to specify advanced options for r limits and k limits and and allow default calculations of these based on the data instead of hard values. Fix default plotting bugs and remember user-defined plot limits. Allow user-defined viewing angle of the morphology.

3   Running the program

There are three steps involved in creating the representative morphology from the data:

1. Open a file.

2. Calculate the Gaussian random field.

3. Create the .pov file.

The Gaussian random field calculation is the most time-intensive step. At the end of this process a 3D binary matrix of solid and pore elements is created in memory. The .pov file is generated from this matrix, enabling the user to create various renderings of the morphology, e.g. 2D and 3D, or to compare and contrast the solid and pore separately.

3.1   Open a file.

The output filename will automatically change to reflect the input filename. It is possible to choose a different output filename. The output files will be saved as tab-delimited ASCII in the same directory as the input files. A log file containing the progression of chosen parameters (namely the q-range and how many data points are included) and the names of files created will also be written. If the log file already exists, it will be appended to; however all subsequent files generated will overwrite the previous ones. An information panel appears advising the user to change the output file name if desired.

Imported files must be tab, space, or comma delimited ASCII files. The background must have been previously subtracted. If exporting ASCII .txt files from Microsoft Excel, please ensure that the file ends at the end of data. Excessive white space at the end of the file will cause problems.

The program assumes the first two columns are q and $I(q)$. Subsequent columns are ignored.

Upon importing a second file, the program will give the user the option to keep the current parameters (porosity, box size and number of divisions, q extrapolation, r and k limits, display options) or revert to default. The former is particularly useful when a series of data is being processed. In both cases the plot limits are reset.

Once the data is successfully imported a plot is shown and the 'Advanced Options', 'Calculate Gaussian Random Field', 'Display Options', 'Plot' and 'Plot Reset' buttons are enabled.
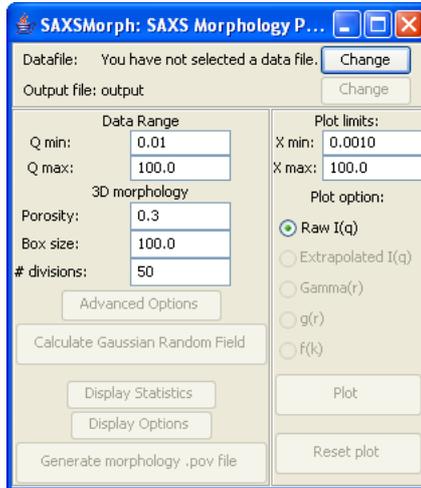
Figure 1: Program control screen upon execution.

3.2 Choose an appropriate q-range.

On the left-hand panel, the values of Qmin and Qmax can be set. These are the limits of the data used in the calculation, shown on the plot by vertical grey lines. The default Qmin and Qmax values are the minimum and maximum q of the data, respectively. These can be changed and the plot updated either by pressing the 'Enter' key, or clicking 'Plot'. The plot limits can also be changed by entering the new value on the right-hand panel and pressing 'Enter' or clicking 'Plot'.

3.3 Calculate the Gaussian Random Field.

3.3.1 Setting the parameters.

On the left-hand panel, enter the porosity (obtained from SEM, for example). A porosity of 0 would be completely solid, and a porosity of 1 would be completely pore. For a two-phase system, 'porosity' is the volume fraction of the second phase. The porosity value can also be calculated from absolute data by using the invariant and knowing the electron density of each phase.

Enter the desired box size. The picture generated is that of a cube with each side the length of the box size. If the input data is in Å^{-1} then the box size units are Å; if the input data is in nm^{-1} then the box size units are nm. It may be worthwhile generating several morphologies of different length scales. Simply change the settings and run the calculation again (remembering to
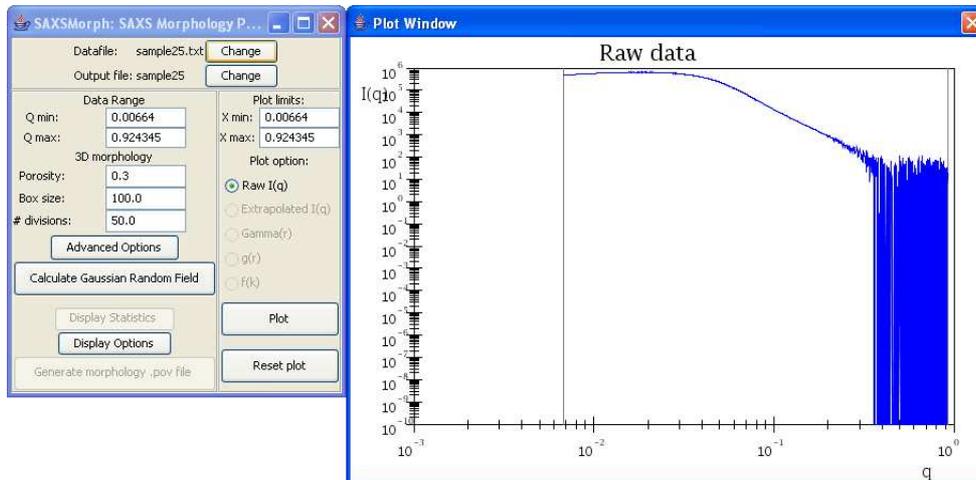
Figure 2: Program control screen after opening a file.

change the output file name). As a general rule of thumb, the box size should be 2-5 times the characteristic length scale (π/q_{peak}).

Enter the desired number of divisions along each side of the box. Please bear in mind that increasing this number increases the number of calculations (and hence the memory required and time the calculation will take) by $O(n^3)$. During the matrix generation step the progress window will update as the matrix is filled. When tested, the limit of the number of divisions was a little over 100 for a PC with 2 GB RAM. In some cases the program is able to detect when the calculation will not have sufficient memory to generate the porosity matrix and display an error message. In other cases the program may hang without such a warning. If this happens, restart the program and decrease the number of divisions.
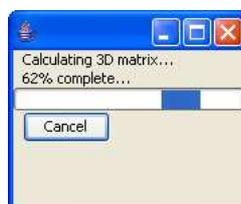

Figure 3: Progress bar during a calculation.

3.3.2 Advanced Options.

If desired, advanced options can be set. These are divided into three panels for setting the $I(q)$ extrapolation parameters, the $\Gamma(r)$ and $g(r)$ data limits, and the $f(k)$ data limits.
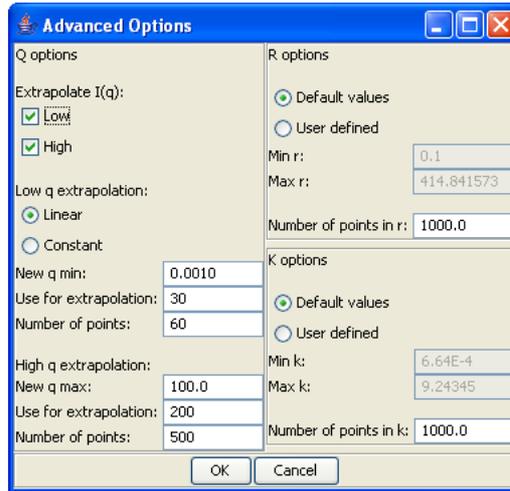

Figure 4: Advanced options panel.

These allow the q extrapolation procedures to be varied:

1. Setting whether extrapolation at high q and/or low q is desired. The default is to extrapolate at both high and low q. The high q region is extrapolated as q^{-4}.

2. Whether to use a linear or constant extrapolation in the low q region. The default is a linear extrapolation; however if the extrapolated values are negative (i.e. unphysical) the program will automatically revert to constant extrapolation. It is not possible to override this.

3. For both the low q and high q extrapolation, to set the new q limit of the extrapolated data, how many points from the data curve to use to calculate the extrapolated curve, and how many points in the extrapolated region. The default parameters are given below.

	Extrapolated limit	Data points used	Extrapolated points
Low q	0.001	30	60
High q	100	200	500

The limits for r and k can also be specified. The lower limits cannot be zero. The default options are:

$$r_{min} = 0.1$$

$$r_{max} = \frac{6.5 \times 5}{\exp\left(\ln(x_{min}) + \ln(x_{max})\right)}$$

$$k_{min} = x_{min} \div 10$$

$$k_{max} = x_{max} \times 10$$

where x_{min} and x_{max} are the minimum and maximum q of the data file.

The default number of points in r and k is 1000; this can be changed independently of selecting the default limits above.

If errors are displayed warning that convergence has not been achieved, increase r_{max} manually.

3.3.3 Calculation.

Clicking 'Calculate Gaussian Random Field' calculates a series of functions, which are plotted when they are available and saved as output files. In order, these are:

- Extrapolated $I(q)$ vs q used to generate the morphology.

- $\Gamma(r)$ vs r.

- $g(r)$ vs r.

- Spectral function, $f(k)$ vs k.

See Section 5 for details on how these are generated, and Section 3.5 for descriptions of the output files. The procedure can be cancelled at any time. Existing plots can be displayed and their limits altered while the calculation is running.

3.3.4 Plotting.

As the calculation proceeds, the plot is updated to show the latest function that has been generated. The plot can be selected by clicking the radio buttons in the right-hand panel. When a function is selected, the plot x limits are automatically set according to the minimum and maximum of the data. These can be changed by entering new values in the 'X min' and 'X max' fields and pressing 'Enter' or clicking the 'Plot' button. These values are remembered as long as the current settings are retained. The default range can be redisplayed for each plot by selecting it and clicking 'Reset Plot'.
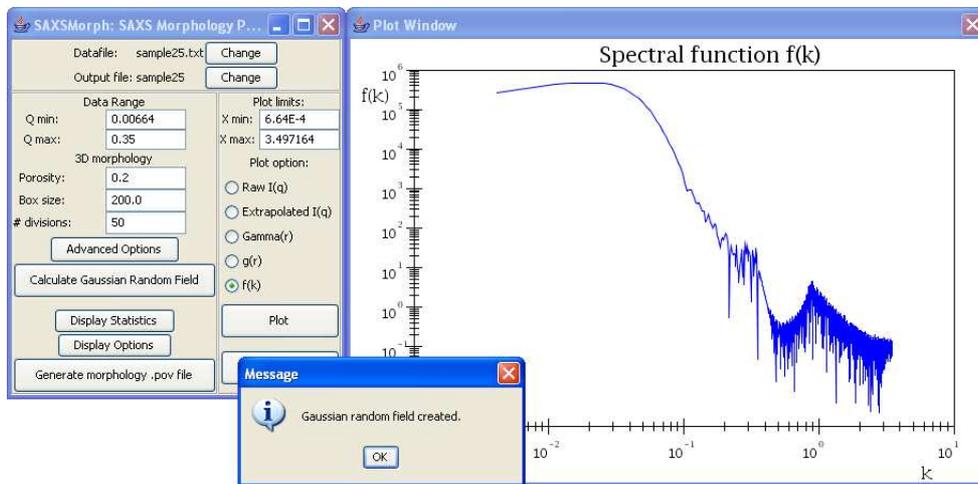

Figure 5: Program upon completion of the Gaussian random field calculation.

3.3.5 Statistics.

Once the Gaussian random field has been created, the 'Display Statistics' button is enabled. When this button is pressed, an information window is displayed giving the surface/volume ratio, whether the morphology is bicontinuous, and the degree of connectivity. These values are also written to the log file. See Section 6 for more information.

3.4 Generate Morphology files.

Once the Gaussian random field has been generated (and stored in internal memory), the 'Display Statistics' and 'Generate morphology' buttons are enabled.
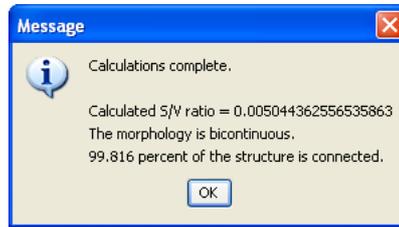
Figure 6: Statistical information display.

It is possible to change the display options by clicking the 'Display Options' button. This allows the user to specify:

- Whether to show the solid or pore.

- If pore, whether to show the largest pore in a different color, specified below.

- Whether to show 3D, 2D single slice, or 2D multiple slices.

- If a 2D slice is selected, which slice to take in the z-direction.

- If a 3D image is selected, what viewing angles θ (around the y-axis) and χ (above horizontal plane).

- Specify the main color and, if 'Largest pore highlighted' is selected, the secondary color.
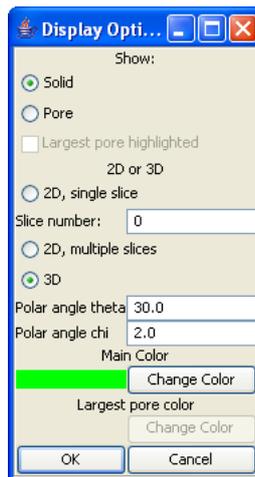

Figure 7: Display options panel.

Once the desired options have been selected (and output file name changed if necessary), clicking 'Generate morphology' will create a .pov file. You will need to download and install POV-Ray separately to visualise the file (www.povray.org).

Animations can be made using the 2D multiple slice feature. This will generate a series of .pov files which need to be rendered, from which an animation can be produced using other software such as Bink & Smacker from RAD Video Tools (for Windows), EasyBMPtoAVI (cross-platform), etc.

If the advanced options are changed after creating the Gaussian random field, the 'Generate morphology' button will be disabled and the Gaussian random field must be re-calculated. If any of the other parameters (Qmin, Qmax, porosity, box size, number of divisions) are changed, a dialog window is displayed giving the user the option whether to re-calculate the Gaussian random field and then the morphology, or use the existing Gaussian random field. In the latter case, the parameters will be changed back to the previous values.
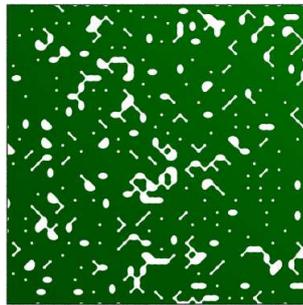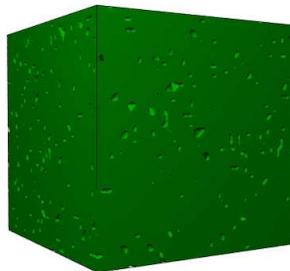

Figure 8: 2D rendering of the solid.

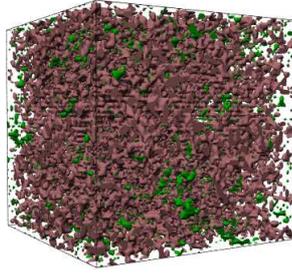Figure 9: 3D rendering of the same solid.

Figure 10: 3D rendering of the pores of the same sample, with the largest pore indicated in pink.

3.5 Output files

For an input file called file.txt, the following output files are written:

file_log.txt	Log file with files written and fitting parameters used. Plain text.
file_newiq.txt	Extrapolated $I(q)$ vs q used to generate the morphology. Two-column ASCII.
file_gamma.txt	$\Gamma(r)$ vs r. Two-column ASCII.
file_gr.txt	$g(r)$ vs r. Two-column ASCII.
file_spec.txt	Spectral function, $f(k)$ vs k. Two-column ASCII.
file.pov	Final POV file. Plain text that can be read and rendered directly by POV-Ray.

4 Availability

The SAXSMorph program and this document are available from
http://www.irl.cri.nz/SAXSfiles

5   Mathematical details

In generating the morphology, the following steps are performed:

Calculate the Debye correlation function, $\Gamma^\alpha(r)$:

$$\Gamma^\alpha(r) = \int_0^\infty 4\pi q^2 I(q) \frac{\sin qr}{qr} dq$$

From this the two-point correlation function $g(r)$ is obtained:

$$\Gamma^\alpha(r) = \Gamma^\alpha(0) - \frac{1}{2\pi} \int_{g(r)}^1 e^{-\frac{\alpha^2}{1+t}} \frac{1}{\sqrt{1-t^2}} dt$$

where $\Gamma^\alpha(0)$ is simply the 'true volume fraction' (1 – porosity) and is related to α by

$$\Gamma^\alpha(0) = \frac{1}{\sqrt{2\pi}} \int_\alpha^\infty e^{-\frac{x^2}{2}} dx$$

Once $g(r)$ is obtained, the spectral function $f(k)$ is calculated:

$$f(k) = \int_0^\infty 4\pi r^2 g(r) \frac{\sin kr}{kr} dr$$

A Gaussian random field is generated by summing many (N = 10000) cosine waves with random phases ϕ_i

$$\Psi(r) = \sqrt{\frac{2}{N}} \sum_1^N \cos(\vec{k}_i \cdot \vec{r} + \phi_i)$$

where the magnitudes of the wavevectors $\vec{k}_i$ are obtained from the spectral function $f(k)$, and the directions are uniformly distributed over a unit sphere[1]. The magnitudes are obtained from the spectral function using the 'rejection method' as follows: Pairs of random numbers are generated to provide co-ordinates which are compared with the graph $f(k)$ vs k. If a point falls below the curve, it is accepted.

In calculating the morphology, a three-dimensional array is constructed and the Gaussian random field at each point compared to α above. If it is less than α, then the point is designated 'solid' and is included in the POV file.

Integrations are calculated using the Romberg method with 2^{10} intervals.

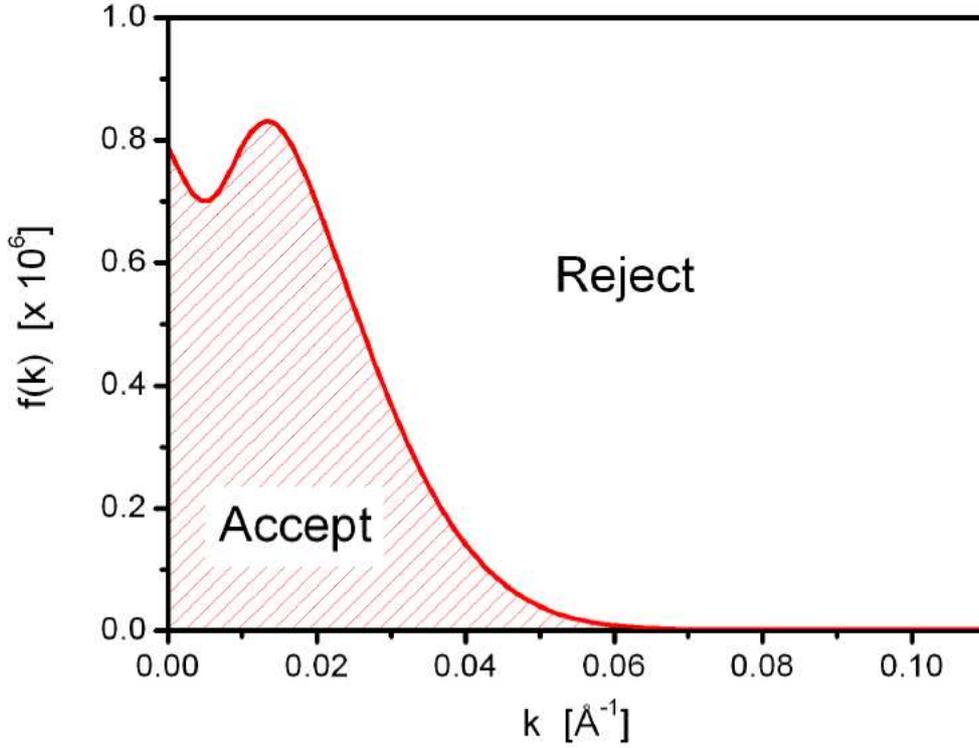[1]N. F. Berk, *Phys. Rev. A* **44** (1991) 5069.

Figure 11: Acceptance criteria.

6 Statistical Calculations

Once the Gaussian random field has been created, the user can press the 'Display Statistics' button to calculate the surface/volume ratio, whether the morphology is bicontinuous, and the degree of connectivity.

6.1 Surface/Volume ratio

This is calculated from I(q) according to[2]

$$\frac{S}{V} = \pi\eta(1-\eta)\frac{\lim\limits_{q\to\infty}\left[I(q)\cdot q^4\right]}{Q} \tag{1}$$

where η is the volume fraction, and Q is the invariant, given by

$$Q = \int_0^\infty I(q)\cdot q^2 dq$$

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[2]O. Glatter and O. Kratky, 'Small Angle X-ray Scattering', Academic Press Inc. (London) 1982, p. 47.

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For this calculation, the extrapolated $I(q)$ is used.

It is easy to see that the numerator in Eq. 6.1 is simply the multiplication factor of the q^{-4} high q extrapolation.

Once again the units are the inverse of the data units (i.e. q in Å^{-1}, S/V in Å; q in nm^{-1}, S/V in nm.

6.2   Bicontinuity

The morphology is bicontinuous if one can find a path through adjacent cells that touches all sides of the box, in both the solid and the pore.

6.3   Degree of Connectivity

This routine counts the number of matrix elements in the largest pore and the largest solid component, and divides this total by the total number of elements in the matrix.

7   Tips

Ensure the data range selected encompasses all features of interest. In particular, if the high q region of the data does not go as q^{-4} (e.g. fractal systems) the minimum feature size in the morphology is limited. The calculated S/V will also be incorrect.

Render the morphology to ensure the correct box size is being used. As a general rule of thumb, the box size should be 2-5 times the characteristic length scale (π/q_{peak}).

Check the $I(q)$ extrapolation goes high enough and the r range for $\Gamma(r)$ (and $g(r)$) is high enough. There is usually some high-frequency oscillation in these and $f(k)$ for $k > q_{max}$ due to infinite integrals being approximated by finite integrals.

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