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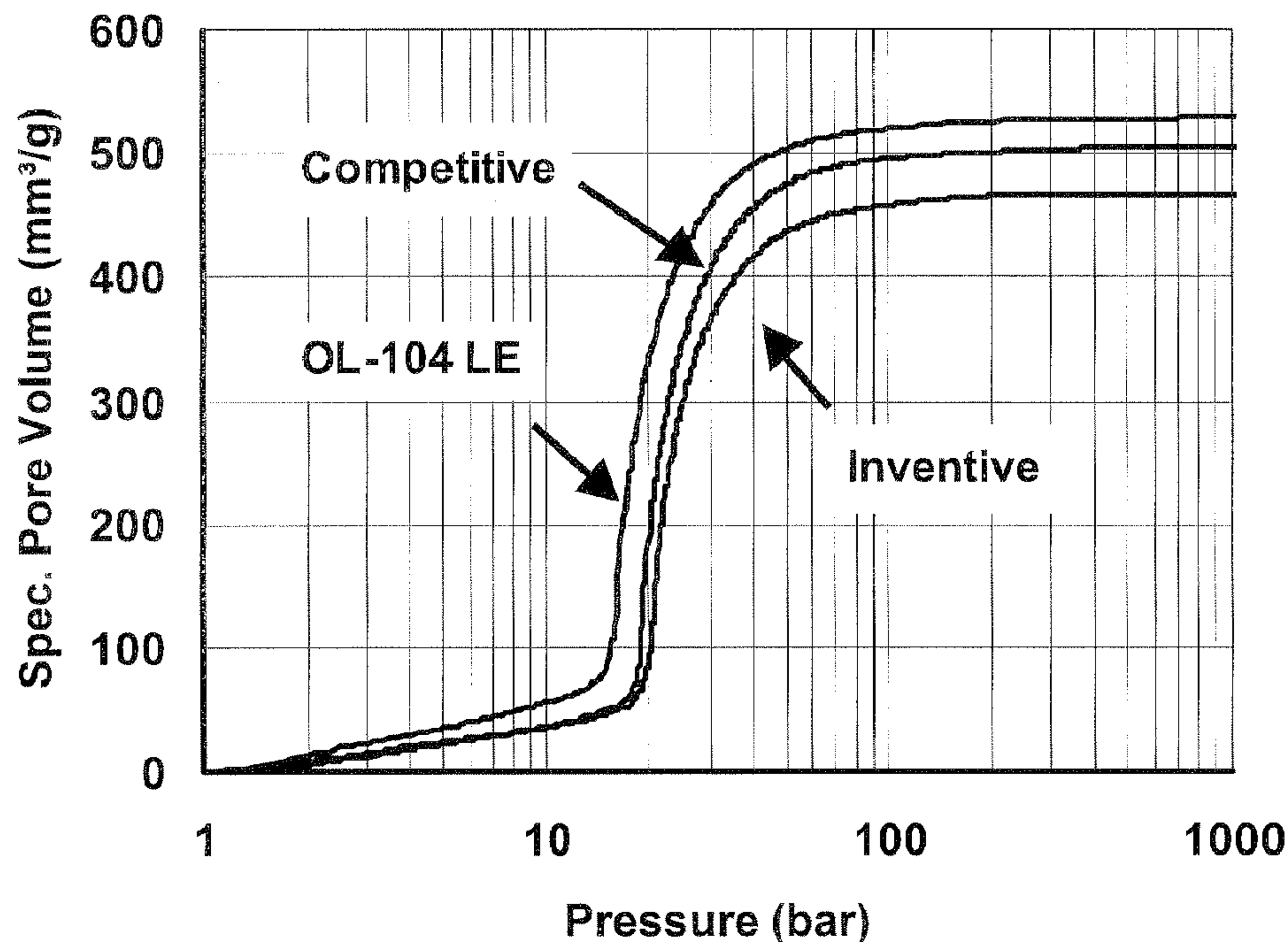
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(54) Title: A PROCESS FOR PRODUCING ALUMINUM HYDROXIDE PARTICLES

Specific pore volume as a function of the applied pressure for the
second intrusion run and the inventive ATH grade in
comparison with standard grades



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A method of after-treating an ATH filter cake by mill-drying, and optionally deagglomeration, to produce flame retardant particles that can be used as a polymer filler.

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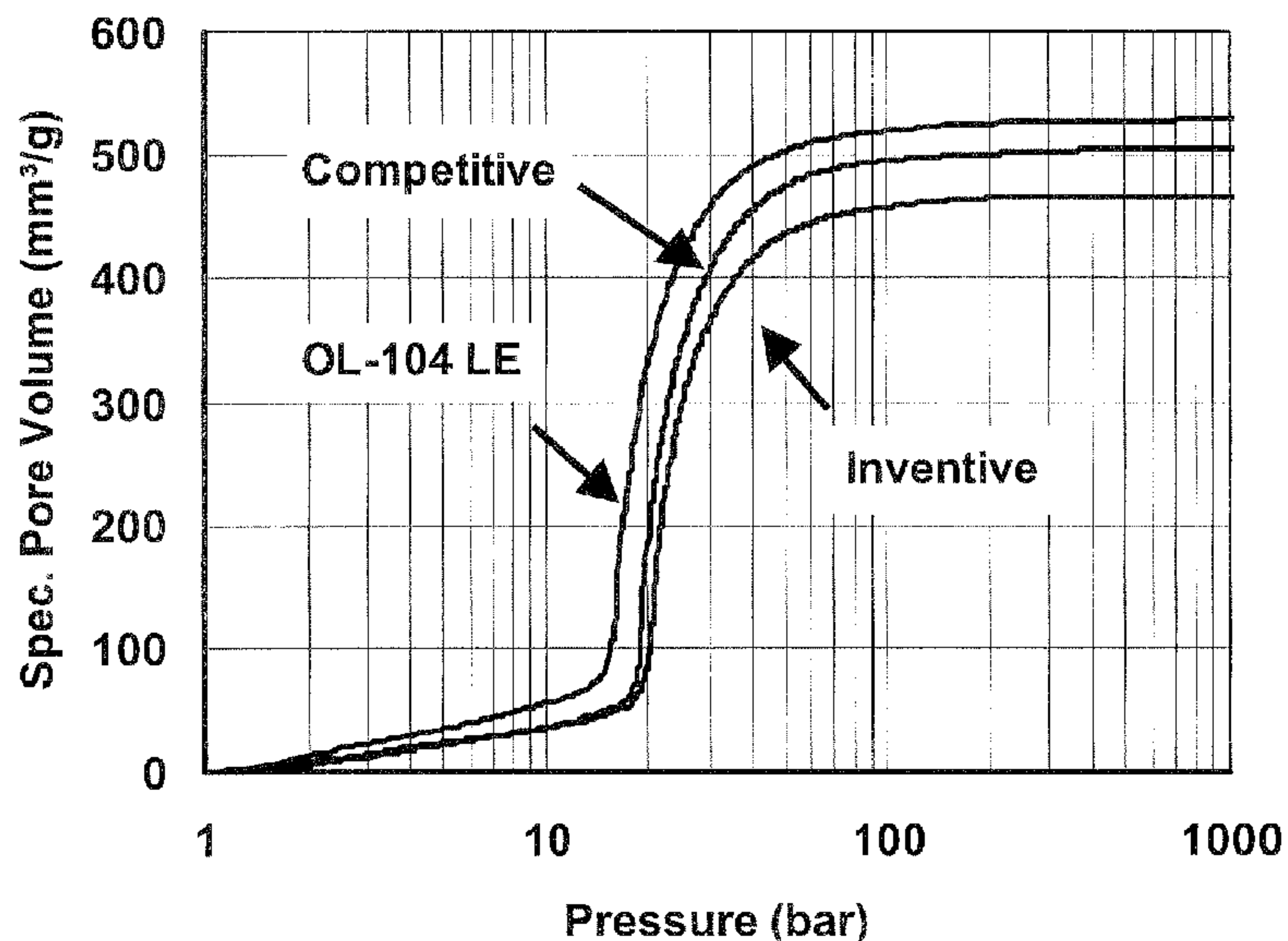
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(54) Title: A PROCESS FOR PRODUCING ALUMINUM HYDROXIDE PARTICLES

Specific pore volume as a function of the applied pressure for the second intrusion run and the inventive ATH grade in comparison with standard grades



(57) Abstract: A method of producing ATH from a filter cake through mill-drying and deagglomeration.

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A PROCESS FOR PRODUCING ALUMINUM HYDROXIDE PARTICLES**FIELD OF THE INVENTION**

[0001] The present invention relates to the production of mineral flame retardants. More particularly the present invention relates to a novel process for the production of aluminum hydroxide flame retardants.

BACKGROUND OF THE INVENTION

[0002] Aluminum hydroxide has a variety of alternative names such as aluminum hydrate, aluminum trihydrate etc., but is commonly referred to as ATH. ATH particles find use as a filler in many materials such as, for example, plastics, rubber, thermosets, papers, etc. These products find use in diverse commercial applications such as wire and cable compounds, conveyor belts, thermoplastics moldings, wall claddings, floorings, etc. ATH is typically used to improve the flame retardancy of such materials and also acts as a smoke suppressant.

[0003] Methods for the synthesis and production of ATH are well known in the art. However, the demand for tailor made ATH grades is increasing, and the current processes are not capable of producing all of these grades. Thus, as the demand for tailor made ATH grades increases, the demand for processes to produce these grades is also increasing.

BRIEF DESCRIPTION OF THE FIGURES

[0004] Figure 1 shows the specific pore volume V as a function of the applied pressure for the second intrusion test run and an ATH according to the present invention ("Inventive"), in comparison with standard grades.

[0005] Figure 2 shows the specific pore volume V plotted against the pore radius r for the second intrusion test run and an ATH according to the present invention ("Inventive"), in comparison with standard grades.

[0006] Figure 3 shows the normalized specific pore volume for an ATH according to the present invention ("Inventive"), in comparison with standard grades, the graph was generated with the maximum specific pore volume for each ATH grade set at 100%, and the other specific volumes of the corresponding ATH grade were divided by this maximum value.

[0007] Figure 4 shows the power draw on the motor of a discharge extruder for the inventive aluminum hydroxide grade used in the Example 1.

[0008] Figure 5 shows the power draw on the motor of a discharge extruder for the comparative aluminum hydroxide grade OL-104 LE used in Example 1.

SUMMARY OF THE INVENTION

[0009] The present invention relates to a process for producing ATH particles comprising:
mill drying a filter moist cake to produce mill dried ATH particles; and

optionally,

deagglomerating the mill-dried ATH particles to produce ATH product particles,

wherein the filter moist cake contains in the range of from about 25 to about 85 wt.%, based on the total weight of the filter moist cake, ATH particles having a d_{50} in the range of from about 1.5 to 3.5 μm , and wherein the ATH product particles have a median pore radius (" r_{50} ") in the range of from about 0.09 to about 0.33 μm .

[0010] In another embodiment, the present invention relates to a process for producing mill-dried ATH particles comprising

mill drying a filter moist cake to produce mill dried ATH particles; and

optionally,

deagglomerating the mill-dried ATH particles to produce ATH product particles,

wherein the filter moist cake contains in the range of from about 25 to about 85 wt.%, based on the total weight of the filter moist cake, ATH particles having a d_{50} in the range of from about 1.5 to 3.5 μm , and wherein the ATH product particles are characterized as having

a) a BET specific surface area of from about 3 to about 6 m^2/g ; and a V_{max} of from about 390 to about 480 mm^3/g ;

or

b) a BET specific surface area of from about 6 to about 9 m^2/g ; and a V_{max} of from about 400 to about 600 mm^3/g ;

or

c) a BET specific surface area of from about 9 to about 15 m^2/g ; and a V_{max} of from about 300 to about 700 mm^3/g .

DETAILED DESCRIPTION OF THE INVENTION

[0011] The inventors hereof believe, while not wishing to be bound by theory, that the wettability of ATH particles with resins depends on the morphology of the ATH particles, and the inventors hereof have unexpectedly discovered that by using the process of the present invention, ATH particles having an improved wettability in relation to ATH particles currently available can be produced. While not wishing to be bound by theory, the inventors hereof believe that this improved wettability is attributable to an improvement in the morphology of the ATH particles produced by the process disclosed herein.

[0012] Again, while not wishing to be bound by theory, the inventors hereof believe that this improved morphology is attributable to the total specific pore volume and/or the median pore radius (" r_{50} ") of the ATH product particles. The inventors hereof believe that, for a given polymer molecule, an ATH product having a higher structured aggregate contains more and

bigger pores and seems to be more difficult to wet, leading to difficulties (higher variations of the power draw on the motor) during compounding in kneaders like Buss Ko-kneaders or twin-screw extruders or other machines known in the art and used to this purpose. Therefore, the inventors hereof have discovered that an ATH filler characterized by smaller median pore sizes and/or lower total pore volumes correlates with an improved wetting with polymeric materials and thus results in improved compounding behavior, i.e. less variations of the power draw of the engines (motors) of compounding machines used to compound a flame retarded resin containing the ATH filler. The inventors hereof have discovered that the process of the present invention is especially well-suited for producing an ATH having these characteristics.

Filter Cake

[0013] In one embodiment of the present invention a filter moist cake, sometimes referred to herein as simply a filter cake, containing ATH particles is mill-dried to produce mill-dried ATH particles. The filter cake typically contains in the range of from about 25 to about 85wt.% ATH particles, based on the total weight of the filter cake. In preferred embodiments, the filter cake contains in the range of from about 40 to about 70 wt.% ATH particles, more preferably in the range of from about 55 to about 65 wt.% ATH particles, both on the same basis.

[0014] In other embodiments, the filter cake contains in the range of from about 40 to about 60 wt.% ATH particles, more preferably in the range of from about 45 to about 55 wt.% ATH particles, both on the same basis. In still other embodiments, the filter cake contains in the range of from about 25 to about 50 wt.% ATH particles, more preferably in the range of from about 30 to about 45 wt.% ATH particles, both on the same basis

[0015] The filter cake used in the practice of the present invention can be obtained from any process used to produce ATH particles. Preferably the filter cake is obtained from a process that involves producing ATH particles through precipitation and filtration. In an exemplary embodiment, the filter cake is obtained from a process that comprises dissolving crude aluminum hydroxide in caustic soda to form a sodium aluminate liquor, which is cooled and filtered thus forming a sodium aluminate liquor useful in this exemplary embodiment. The sodium aluminate liquor thus produced typically has a molar ratio of Na_2O to Al_2O_3 in the range of from about 1.4:1 to about 1.55:1. In order to precipitate ATH particles from the sodium aluminate liquor, ATH seed particles are added to the sodium aluminate liquor in an amount in the range of from about 1 g of ATH seed particles per liter of sodium aluminate liquor to about 3 g of ATH seed particles per liter of sodium aluminate liquor thus forming a

process mixture. The ATH seed particles are added to the sodium aluminate liquor when the sodium aluminate liquor is at a liquor temperature of from about 45 to about 80°C. After the addition of the ATH seed particles, the process mixture is stirred for about 100 h or alternatively until the molar ratio of Na_2O to Al_2O_3 is in the range of from about 2.2 : 1 to about 3.5 : 1, thus forming an ATH suspension. The obtained ATH suspension typically comprises from about 80 to about 160 g/l ATH, based on the suspension. However, the ATH concentration can be varied to fall within the ranges described above. The obtained ATH suspension is then filtered and washed to remove impurities therefrom, thus forming a filter cake. Before the filter cake is mill-dried, it can be washed one, or in some embodiments more than one, times with water, preferably de-salted water.

[0016] In some embodiments, the ATH particles in the filter cake are generally characterized as having a BET in the range of from about 1.0 to about 4.0 m^2/g . In preferred embodiments, the ATH particles in the filter cake have a BET in the range of from about 1.5 to about 2.5 m^2/g . The ATH particles in the filter cake can also be further characterized as having a d_{50} in the range of from about 1.8 to about 3.5 μm . In preferred embodiments, the ATH particles in the filter cake have a d_{50} in the range of from about 1.8 to about 2.5 μm , which is coarser than the ATH product particles produced by the present invention.

[0017] In other embodiments, the ATH particles in the filter cake are characterized as having a BET in the range of from about 4.0 to about 8.0 m^2/g . In preferred embodiments, the ATH particles in the filter cake have a BET in the range of from about 5 to about 7 m^2/g . The ATH particles in the filter cake can be further characterized as having a d_{50} in the range of from about 1.5 to about 2.5 μm . In preferred embodiments, the ATH particles in the filter cake have a d_{50} in the range of from about 1.6 to about 2.0 μm , which is coarser than the ATH product particles produced by the present invention.

[0018] In still other embodiments, the ATH particles in the filter cake are characterized as having a BET in the range of from about 8.0 to about 14 m^2/g . In preferred embodiments, the ATH particles in the filter cake have a BET in the range of from about 9 to about 12 m^2/g . The ATH particles in the filter cake can be further characterized as having a d_{50} in the range of from about 1.5 to about 2.0 μm . In preferred embodiments, the ATH particles in the filter cake have a d_{50} in the range of from about 1.5 to about 1.8 μm , which is coarser than the ATH product particles produced by the present invention.

[0019] By coarser than the ATH product particles, it is meant that the upper limit of the d_{50} value of the ATH particles in the filter cake is generally at least about 0.2 μm higher than the upper limit of the d_{50} of the ATH product particles produced by the present invention.

[0020] The inventors hereof, while not wishing to be bound by theory, believe that the improved morphology of the ATH product particles produced by the present invention is at least partially attributable to the process used to precipitate the ATH. Thus, while mill-drying techniques are known in the art, the inventors hereof have discovered that by using the precipitation and filtration processes described herein, including preferred embodiments, along with the mill-drying process described herein, ATH product particles having improved morphology, as described below, can be readily produced.

Mill-Drying

[0021] As discussed above, the present invention involves mill-drying a filter cake to produce mill-dried ATH particles that are optionally subjected to deagglomeration. "Mill drying" and "mill dried" as used herein, is meant that the filter cake, is dried in a turbulent hot air-stream in a mill drying unit. The mill-drying unit comprises a rotor that is firmly mounted on a solid shaft that rotates at a high circumferential speed. The rotational movement in connection with a high air through-put converts the through-flowing hot air into extremely fast air vortices which take up the mixture to be dried, accelerate it, and distribute and dry the mixture. After having been dried completely, the dry-milled ATH particles are transported via the turbulent air out of the mill and separated from the hot air and vapors by using conventional filter systems. In another embodiment of the present invention, after having been dried completely, the dry-milled ATH particles are transported via the turbulent air through an air classifier which is integrated into the mill, and are then transported via the turbulent air out of the mill and separated from the hot air and vapors by using conventional filter systems.

[0022] The throughput of the hot air used to dry the filter cake is typically greater than about 3,000 Bm³/h, preferably greater than about 5,000 Bm³/h, more preferably from about 3,000 Bm³/h to about 40,000 Bm³/h, and most preferably from about 5,000 Bm³/h to about 30,000 Bm³/h.

[0023] In order to achieve throughputs this high, the rotor of the mill drying unit typically has a circumferential speed of greater than about 40 m/sec, preferably greater than about 60 m/sec, more preferably greater than 70 m/sec, and most preferably in a range of about 70 m/sec to about 140 m/sec. The high rotational speed of the motor and high throughput of hot air results in the hot air stream having a Reynolds number greater than about 3,000.

[0024] The temperature of the hot air stream used to mill dry the filter cake is generally greater than about 150°C, preferably greater than about 270°C. In a more preferred

embodiment, the temperature of the hot air stream is in the range of from about 150°C to about 550°C, most preferably in the range of from about 270°C to about 500°C.

[0025] The mill-drying of the filter cake produces mill-dried ATH particles that have a larger BET specific surface area, as determined by DIN-66132, than the starting ATH particles in the filter cake. Typically, the BET of the mill-dried ATH are more than about 10% greater than the ATH particles in the filter cake. Preferably the BET of the mill-dried ATH is in the range of from about 10% to about 40% greater than the ATH particles in the filter cake. More preferably the BET of the mill-dried ATH particles is in the range of from about 10% to about 25% greater than the ATH particles in the filter cake.

[0026] The mill-dried ATH particles thus produced can be used “as is” in many applications. However, in some embodiments, the mill-dried ATH particles are further processed to reduce, or in some embodiments eliminate, agglomerates. Agglomerates are common in ATH particle production processes, and their presence can, and in some applications does, deleteriously affect the performance of the ATH particles in a resin. Therefore, the reduction, preferably elimination, of agglomerates is highly desired by ATH producers.

[0027] In the practice of the present invention, the number of agglomerates, or degree of agglomeration, present in the mill-dried ATH particles can be reduced by subjecting the mill-dried ATH particles to a further deagglomeration processing step.

Deagglomeration

[0028] By deagglomeration, it is meant that the mill-dried ATH particles are subjected to a further treatment wherein the number of agglomerates, or degree of agglomeration, present in the mill-dried ATH particles are reduced (i.e. the number of agglomerates present in the mill-dried ATH particles is greater than the number of agglomerates present in the ATH product particles), in some embodiments substantially eliminated, with little reduction in the particle size of the mill-dried ATH. By “little particle size reduction” it is meant that the d_{50} of the ATH product particles is greater than or equal to 90% of the mill-dried ATH particles. The rest of the properties of the mill-dried ATH particles are the same or substantially the same as the ATH product particles produced from deagglomerating the mill-dried ATH particles. In preferred embodiments, the d_{50} of the dry-milled ATH is in the range of from about 90% to about 95% of the mill-dried ATH particles, more preferably within the range of from about 95% to about 99% of the mill-dried ATH particles.

[0029] The reduction in the agglomerates present in the mill-dried ATH particles can be achieved by using any technique known to be effective at reducing agglomerates. In preferred embodiments, the deagglomeration is achieved through the use of air classifiers or

pin mills. In some embodiments, deagglomeration is achieved through the use of one or more pin mills, in other embodiments, one or more air classifiers.

[0030] Air classifiers suitable for use herein include those using gravitational forces, centrifugal forces, inertial forces, or any combination thereof, to classify the ATH product particles. The use of these classifiers is well known in the art, and one having ordinary skill in the art and knowledge of the desired final ATH product size can readily select classifiers containing suitable screens and/or sieves.

[0031] Pin Mills suitable for use herein include dry and wet pin mills. As with air classifiers, the use of pin mills is well known in the art, and one having ordinary skill in the art and knowledge of the desired final ATH product particles properties can readily select the best pin mill to fit a particular application.

Improved Morphology ATH Product Particles

[0032] In general, the process of the present invention can be used to produce ATH product particles having an oil absorption, as determined by ISO 787-5:1980 of in the range of from about 1 to about 35%, a BET specific surface area, as determined by DIN-66132, in the range of from about 1 to 15 m²/g, and a d₅₀ in the range of from about 0.5 to 2.5 μm.

[0033] However, the process of the present invention is especially well-suited to produce ATH product particles having an improved morphology when compared with currently available ATH particles. Again, while not wishing to be bound by theory, the inventors hereof believe that this improved morphology is attributable to the total specific pore volume and/or the median pore radius ("r₅₀") of the ATH product particles produced herein. The inventors hereof believe that, for a given polymer molecule, an ATH having a higher structured aggregate contains more and bigger pores and seems to be more difficult to wet, leading to difficulties (higher variations of the power draw on the motor) during compounding in kneaders like Buss Ko-kneaders or twin-screw extruders or other machines known in the art and used to this purpose. The inventors hereof have discovered that the process of the present invention produces ATH product particles characterized by smaller median pore sizes and/or lower total pore volumes, when compared to currently available ATH, which correlates to an improved wetting of the ATH product particles with polymeric materials, and thus, results in improved compounding behavior, i.e. less variations of the power draw of the engines (motors) of compounding machines used to compound a flame retarded resin containing the ATH product particle filler, when compared to currently available ATH particles.

[0034] The r_{50} and the specific pore volume at about 1000 bar (“ $V_{\max}$ ”) of the ATH product particles produced by the present invention can be derived from mercury porosimetry. The theory of mercury porosimetry is based on the physical principle that a non-reactive, non-wetting liquid will not penetrate pores until sufficient pressure is applied to force its entrance. Thus, the higher the pressure necessary for the liquid to enter the pores, the smaller the pore size. A smaller pore size and/or a lower total specific pore volume were found to correlate to better wettability of the ATH product particles produced by the present invention. The pore size of the ATH product particles produced by the present invention can be calculated from data derived from mercury porosimetry using a Porosimeter 2000 from Carlo Erba Strumentazione, Italy. According to the manual of the Porosimeter 2000, the following equation is used to calculate the pore radius r from the measured pressure p : $r = -2 \gamma \cos(\theta)/p$; wherein θ is the wetting angle and γ is the surface tension. The measurements taken herein used a value of 141.3° for θ and γ was set to 480 dyn/cm.

[0035] In order to improve the repeatability of the measurements, the pore size of the ATH product particles was calculated from the second ATH intrusion test run, as described in the manual of the Porosimeter 2000. The second test run was used because the inventors observed that an amount of mercury having the volume V_0 remains in the sample of ATH particles after extrusion, i.e. after release of the pressure to ambient pressure. Thus, the r_{50} can be derived from this data as explained below with reference to Figures 1, 2, and 3.

[0036] In the first test run, a sample of ATH product particles produced by the present invention was prepared as described in the manual of the Porosimeter 2000, and the pore volume was measured as a function of the applied intrusion pressure p using a maximum pressure of 1000 bar. The pressure was released and allowed to reach ambient pressure upon completion of the first test run. A second intrusion test run (according to the manual of the Porosimeter 2000) utilizing the same ATH sample, unadulterated, from the first test run was performed, where the measurement of the specific pore volume $V(p)$ of the second test run takes the volume V_0 as a new starting volume, which is then set to zero for the second test run.

[0037] In the second intrusion test run, the measurement of the specific pore volume $V(p)$ of the sample was again performed as a function of the applied intrusion pressure using a maximum pressure of 1000 bar. Figure 1 shows the specific pore volume V as a function of the applied pressure for the second intrusion test run and an ATH, grade no. 1, produced according to the present invention in comparison with current commercially available ATH

products. The pore volume at about 1000 bar, i.e. the maximum pressure used in the measurement, is referred to as $V_{\max}$ herein.

[0038] From the second ATH intrusion test run, the pore radius r was calculated by the Porosimeter 2000 according to the formula $r = -2 \gamma \cos(\theta)/p$; wherein θ is the wetting angle, γ is the surface tension and p the intrusion pressure. For all r -measurements taken herein, a value of 141.3° for θ was used and γ was set to 480 dyn/cm. The specific pore volume can thus be plotted against the pore radius r . Figure 2 shows the specific pore volume V of the second intrusion test run (using the same sample) plotted against the pore radius r .

[0039] Figure 3 shows the normalized specific pore volume of the second intrusion test run plotted against the pore radius r , i.e. in this curve, the maximum specific pore volume of the second intrusion test run, $V_{\max}$, was set to 100% and the other specific volumes for that particular ATH were divided by this maximum value. The pore radius at 50% of the relative specific pore volume, by definition, is called median pore radius r_{50} herein. For example, according to Figure 3, the median pore radius r_{50} for an ATH according to the present invention, i.e. Inventive, is $0.33\mu\text{m}$.

[0040] The procedure described above was repeated using samples of ATH product particles produced according to the present invention, and the ATH product particles produced by the present invention were found to have an r_{50} , i.e. a pore radius at 50% of the relative specific pore volume, in the range of from about 0.09 to about $0.33\mu\text{m}$. In preferred embodiments of the present invention, the r_{50} of the ATH product particles produced by the present invention is in the range of from about 0.20 to about $0.33\mu\text{m}$, more preferably in the range of from about 0.2 to about $0.3\mu\text{m}$. In other preferred embodiments, the r_{50} is in the range of from about 0.185 to about $0.325\mu\text{m}$, more preferably in the range of from about 0.185 to about $0.25\mu\text{m}$. In still other preferred embodiments, the r_{50} is in the range of from about 0.09 to about $0.21\mu\text{m}$, more preferably in the range of from about 0.09 to about $0.165\mu\text{m}$.

[0041] The ATH product particles produced by the present invention can also be characterized as having a $V_{\max}$, i.e. maximum specific pore volume at about 1000 bar, in the range of from about 300 to about $700\text{ mm}^3/\text{g}$. In preferred embodiments of the present invention, the $V_{\max}$ of the ATH product particles produced by the present invention is in the range of from about 390 to about $480\text{ mm}^3/\text{g}$, more preferably in the range of from about 410 to about $450\text{ mm}^3/\text{g}$. In other preferred embodiments, the $V_{\max}$ is in the range of from about 400 to about $600\text{ mm}^3/\text{g}$, more preferably in the range of from about 450 to about $550\text{ mm}^3/\text{g}$. In yet other preferred embodiments, the $V_{\max}$ is in the range of from about 300 to about $700\text{ mm}^3/\text{g}$, more preferably in the range of from about 350 to about $550\text{ mm}^3/\text{g}$.

[0042] The ATH product particles produced by the present invention can also be characterized as having an oil absorption, as determined by ISO 787-5:1980 of in the range of from about 1 to about 35%. In some preferred embodiments, the ATH product particles produced by the present invention are characterized as having an oil absorption in the range of from about 23 to about 30%, more preferably in the range of from about 25% to about 28%. In other preferred embodiments, the ATH product particles produced by the present invention are characterized as having an oil absorption in the range of from about 25% to about 32%, more preferably in the range of from about 26% to about 30%. In yet other preferred embodiments, the ATH product particles produced by the present invention are characterized as having an oil absorption in the range of from about 25 to about 35% more preferably in the range of from about 27% to about 32%. In other embodiments, the oil absorption of the ATH product particles produced by the present invention are in the range of from about 19% to about 23%, and in still other embodiments, the oil absorption of the ATH product particles produced by the present invention is in the range of from about 21% to about 25%.

[0043] The ATH product particles produced by the present invention can also be characterized as having a BET specific surface area, as determined by DIN-66132, in the range of from about 1 to 15 m²/g. In preferred embodiments, the ATH product particles produced by the present invention have a BET specific surface in the range of from about 3 to about 6 m²/g, more preferably in the range of from about 3.5 to about 5.5 m²/g. In other preferred embodiments, the ATH product particles produced by the present invention have a BET specific surface of in the range of from about 6 to about 9 m²/g, more preferably in the range of from about 6.5 to about 8.5 m²/g. In still other preferred embodiments, the ATH product particles produced by the present invention have a BET specific surface in the range of from about 9 to about 15 m²/g, more preferably in the range of from about 10.5 to about 12.5 m²/g.

[0044] The ATH product particles produced by the present invention can also be characterized as having a d₅₀ in the range of from about 0.5 to 2.5 μm. In preferred embodiments, the ATH product particles produced by the present invention have a d₅₀ in the range of from about 1.5 to about 2.5 μm, more preferably in the range of from about 1.8 to about 2.2 μm. In other preferred embodiments, the ATH product particles produced by the present invention have a d₅₀ in the range of from about 1.3 to about 2.0 μm, more preferably in the range of from about 1.4 to about 1.8 μm. In still other preferred embodiments, the

ATH product particles produced by the present invention have a d_{50} in the range of from about 0.9 to about 1.8 μm , more preferably in the range of from about 1.1 to about 1.5 μm .

[0045] It should be noted that all particle diameter measurements, i.e. d_{50} , disclosed herein were measured by laser diffraction using a Cilas 1064 L laser spectrometer from Quantachrome. Generally, the procedure used herein to measure the d_{50} , can be practiced by first introducing a suitable water-dispersant solution (preparation see below) into the sample-preparation vessel of the apparatus. The standard measurement called "Particle Expert" is then selected, the measurement model "Range 1" is also selected, and apparatus-internal parameters, which apply to the expected particle size distribution, are then chosen. It should be noted that during the measurements the sample is typically exposed to ultrasound for about 60 seconds during the dispersion and during the measurement. After a background measurement has taken place, from about 75 to about 100 mg of the sample to be analyzed is placed in the sample vessel with the water/dispersant solution and the measurement started. The water/dispersant solution can be prepared by first preparing a concentrate from 500 g Calgon, available from KMF Laborchemie, with 3 liters of CAL Polysalt, available from BASF. This solution is made up to 10 liters with deionized water. 100 ml of this original 10 liters is taken and in turn diluted further to 10 liters with deionized water, and this final solution is used as the water-dispersant solution described above.

Use of the ATH Product Particles as a Flame Retardant

[0046] The ATH product particles produced according to the present invention can be used as a flame retardant in a variety of synthetic resins. Non-limiting examples of thermoplastic resins where the dry-milled ATH particles find use include polyethylene, ethylene-propylene copolymer, polymers and copolymers of C_2 to C_8 olefins (α -olefin) such as polybutene, poly(4-methylpentene-1) or the like, copolymers of these olefins and diene, ethylene-acrylate copolymer, polystyrene, ABS resin, AAS resin, AS resin, MBS resin, ethylene-vinyl chloride copolymer resin, ethylene-vinyl acetate copolymer resin, ethylene-vinyl chloride-vinyl acetate graft polymer resin, vinylidene chloride, polyvinyl chloride, chlorinated polyethylene, vinyl chloride-propylene copolymer, vinyl acetate resin, phenoxy resin, and the like. Further examples of suitable synthetic resins include thermosetting resins such as epoxy resin, phenol resin, melamine resin, unsaturated polyester resin, alkyd resin and urea resin and natural or synthetic rubbers such as EPDM, butyl rubber, isoprene rubber, SBR, NIR, urethane rubber, polybutadiene rubber, acrylic rubber, silicone rubber, fluoro-elastomer, NBR and chloro-sulfonated polyethylene are also included. Further included are polymeric suspensions (latices).

[0047] Preferably, the synthetic resin is a polyethylene-based resins such as high-density polyethylene, low-density polyethylene, linear low-density polyethylene, ultra low-density polyethylene, EVA (ethylene-vinyl acetate resin), EEA (ethylene-ethyl acrylate resin), EMA (ethylene-methyl acrylate copolymer resin), EAA (ethylene-acrylic acid copolymer resin) and ultra high molecular weight polyethylene; and polymers and copolymers of C₂ to C₈ olefins (α -olefin) such as polybutene and poly(4-methylpentene-1), polyvinyl chloride and rubbers. In a more preferred embodiment, the synthetic resin is a polyethylene-based resin.

[0048] The inventors have discovered that by using the ATH product particles according to the present invention as flame retardants in synthetic resins, better compounding performance, of the aluminum hydroxide containing synthetic resin can be achieved. The better compounding performance is highly desired by those compounders, manufactures, etc. producing highly filled flame retarded compounds and final extruded or molded articles out of ATH-product-particle-containing synthetic resins. By highly filled, it is meant those containing the flame retarding amount of ATH product particles, discussed below.

[0049] By better compounding performance, it is meant that variations in the amplitude of the energy level of compounding machines like Buss Ko-kneaders or twin screw extruders needed to mix a synthetic resin containing ATH product particles produced according to the present invention are smaller than those of compounding machines mixing a synthetic resin containing conventional ATH particles. The smaller variations in the energy level allows for higher throughputs of the ATH-product-particle-containing synthetic resins to be mixed or extruded and/or a more uniform (homogenous) material.

[0050] Thus, in one embodiment, the present invention relates to a flame retarded polymer formulation comprising at least one synthetic resin, selected from those described above, in some embodiments only one and a flame retarding amount of ATH product particles produced according to the present invention, and extruded and/or molded article made from the flame retarded polymer formulation.

[0051] By a flame retarding amount of the ATH product particles, it is generally meant in the range of from about 5 wt% to about 90 wt%, based on the weight of the flame retarded polymer formulation, and more preferably from about 20 wt% to about 70 wt%, on the same basis. In a most preferred embodiment, a flame retarding amount is from about 30 wt% to about 65 wt% of the ATH product particles, on the same basis.

[0052] The flame retarded polymer formulation can also contain other additives commonly used in the art. Non-limiting examples of other additives that are suitable for use in the flame retarded polymer formulations of the present invention include extrusion aids such as

polyethylene waxes, Si-based extrusion aids, fatty acids; coupling agents such as amino-, vinyl- or alkyl silanes or maleic acid grafted polymers; sodium stearate or calcium stearate; organoperoxides; dyes; pigments; fillers; blowing agents; deodorants; thermal stabilizers; antioxidants; antistatic agents; reinforcing agents; metal scavengers or deactivators; impact modifiers; processing aids; mold release aids, lubricants; anti-blocking agents; other flame retardants; UV stabilizers; plasticizers; flow aids; and the like. If desired, nucleating agents such as calcium silicate or indigo can be included in the flame retarded polymer formulations also. The proportions of the other optional additives are conventional and can be varied to suit the needs of any given situation.

[0053] The methods of incorporation and addition of the components of the flame-retarded polymer formulation is conducted is not critical to the present invention and can be any known in the art so long as the method selected involves substantially uniform mixing. For example, each of the above components, and optional additives if used, can be mixed using a Buss Ko-kneader, internal mixers, Farrel continuous mixers or twin screw extruders or in some cases also single screw extruders or two roll mills. The flame retarded polymer formulation can then be molded in a subsequent processing step, if so desired. In some embodiments, apparatuses can be used that thoroughly mix the components to form the flame retarded polymer formulation and also mold an article out of the flame retarded polymer formulation. Further, the molded article of the flame-retardant polymer formulation may be used after fabrication for applications such as stretch processing, emboss processing, coating, printing, plating, perforation or cutting. The molded article may also be affixed to a material other than the flame-retardant polymer formulation of the present invention, such as a plasterboard, wood, a block board, a metal material or stone. However, the kneaded mixture can also be inflation-molded, injection-molded, extrusion-molded, blow-molded, press-molded, rotation-molded or calender-molded.

[0054] In the case of an extruded article, any extrusion technique known to be effective with the synthetic resins mixture described above can be used. In one exemplary technique, the synthetic resin, aluminum hydroxide particles, and optional components, if chosen, are compounded in a compounding machine to form a flame-retardant resin formulation as described above. The flame-retardant resin formulation is then heated to a molten state in an extruder, and the molten flame-retardant resin formulation is then extruded through a selected die to form an extruded article or to coat for example a metal wire or a glass fiber used for data transmission.

[0055] The above description is directed to several embodiments of the present invention. Those skilled in the art will recognize that other means, which are equally effective, could be devised for carrying out the spirit of this invention. It should also be noted that preferred embodiments of the present invention contemplate that all ranges discussed herein include ranges from any lower amount to any higher amount. For example, when discussing the oil absorption of the dry-milled ATH particles, it is contemplated that ranges from about 30% to about 32%, about 19% to about 25%, about 21% to about 27%, etc. are within the scope of the present invention.

[0056] The following examples will illustrate the present invention, but are not meant to be limiting in any manner.

EXAMPLES

[0057] The r_{50} and $V_{\max}$ described in the examples below was derived from mercury porosimetry using a Porosimeter 2000, as described above. All d_{50} , BET, oil absorption, etc., unless otherwise indicated, were measured according to the techniques described above. Also, the term “Inventive ATH” as used in the examples is meant to refer to an ATH produced according to the present invention, and “Comparative ATH”, “Competitive”, and “Comparative” is meant to refer to an ATH that is commercially available and not produced according to the present invention.

EXAMPLE 1

[0058] An ATH filter cake with a solid content of 50 wt.% was directly fed to a drying mill with a rate of 280 l/h. The aluminum hydroxide in the filter cake, prior to dry milling, had a BET specific surface area of 3.0 m²/g and a median particle size of 2.38 μm. The mill was operated under conditions that included an air flow rate of between 3000 – 3500 Bm³/h at a temperature of 400- 450°C and a rotor speed of 55 m/s.

[0059] After milling, the mill-dried aluminum hydroxide particles were collected from the hot air stream via an air filter system. The product properties of the recovered aluminum hydroxide particles are contained in Table 1, below.

[0060] The product properties of the comparative aluminum hydroxide grade Martinal OL-104 LE produced by Martinswerk GmbH and the product properties of a competitive aluminum hydroxide grade “Competitive” are also shown in Table 1.

TABLE 1				
	Median pore radius ("r₅₀") (μm)	Maximum specific pore volume V_{max} (mm³/g)	Median particle size d₅₀ (μm)	Specific BET surface (m²/g)
Comparative ATH OL-104 LE	0.419	529	1.83	3.2
Competitive	0.353	504	1.52	3.2
Inventive ATH	0.33	466	1.80	3.7

[0061] As can be seen in Table 1, the inventive aluminum hydroxide grade, an ATH produced according to the present invention, has the lowest median pore radius and the lowest maximum specific pore volume.

EXAMPLE 2

[0062] The comparative aluminum hydroxide particles Martinal OL-104 LE and the inventive aluminum hydroxide grade of Example 1 were separately used to form a flame-retardant resin formulation. The synthetic resin used was a mixture of EVA Escorene® Ultra UL00328 from ExxonMobil together with a LLDPE grade LL1001XV from ExxonMobil, Ethanox® 310 antioxidant available commercially from the Albemarle® Corporation, and an amino silane Dynasylan AMEO from Degussa. The components were mixed on a 46 mm Buss Ko-kneader (L/D ratio = 11) at a throughput of 25 kg/h with temperature settings and screw speed chosen in a usual manner familiar to a person skilled in the art. The amount of each component used in formulating the flame-retardant resin formulation is detailed in Table 2, below.

Table 2	
	Phr (parts per hundred total resin)
Escorene Ultra UL00328	80
LL1001XV	20
Aluminum hydroxide	150
AMEO silane	1.6
Ethanox 310	0.6

[0063] In forming the flame-retardant resin formulation, the AMEO silane and Ethanox® 310 were first blended with the total amount of synthetic resin in a drum prior to Buss compounding. By means of loss in weight feeders, the resin/silane/antioxidant blend was fed into the first inlet of the Buss kneader, together with 50 % of the total amount of aluminum

hydroxide, and the remaining 50% of the aluminum hydroxide was fed into the second feeding port of the Buss kneader. The discharge extruder was flanged perpendicular to the Buss Ko-kneader and had a screw size of 70 mm. Figure 4 shows the power draw on the motor of the discharge extruder for the inventive aluminum hydroxide grade no. 1. Figure 5 shows the power draw on the motor of the discharge extruder for the comparative aluminum hydroxide grade OL-104 LE, produced by Nartinswerk GmbH.

[0064] As demonstrated in Figures 4 and 5, variations in the energy (power) draw of the discharge extruder are significantly reduced when the aluminum hydroxide particles according to the present invention are used in the flame-retardant resin formulation. As stated above, smaller variations in energy level allows for higher throughputs and/or a more uniform (homogenous) flame-retardant resin formulation.

WHAT IS CLAIMED:

1. A process for producing ATH particles comprising:
 - a) mill drying a filter moist cake to produce mill dried ATH particles; and optionally
 - b) reducing the number of any agglomerates present in said mill-dried ATH particles thereby producing ATH product particles,

wherein the filter moist cake contains in the range of from about 25 to about 85 wt.% ATH particles, based on the total weight of the filter moist cake, having a d_{50} in the range of from about 1.5 to about 3.5 μm , and wherein the ATH product particles and/or the mill-dried ATH particles have a median pore radius (" r_{50} ") in the range of from about 0.09 to about 0.33 μm and/or a V_{max} in the range of from about 300 to about 700 mm^3/g .
2. The process according to claim 1 wherein said ATH product particles and/or the mill-dried ATH particles have:
 - a) BET specific surface area of from about 3 to about 6 m^2/g ; and a V_{max} in the range of from about 390 to about 480 mm^3/g ;

or

 - b) a BET specific surface area of from about 6 to about 9 m^2/g ; and a V_{max} in the range from about 400 to about 600 mm^3/g

or

 - c) a BET specific surface area of from about 9 to about 15 m^2/g ; and a V_{max} in the range from about 300 to about 700 mm^3/g .
3. The process according to claim 1 wherein said filter moist cake is obtained from a process that involves producing ATH particles through precipitation and filtration.
4. The process according to claim 2 wherein said filter moist cake is obtained from a process that comprises dissolving aluminum hydroxide in caustic soda to form a sodium aluminate liquor; filtering the sodium aluminate solution to remove impurities; cooling and diluting the sodium aluminate liquor to an appropriate temperature and concentration; adding ATH seed particles to the sodium aluminate solution; allowing ATH particles to precipitate from the solution thus forming an ATH suspension containing in the range of from about 80 to about 160 g/l ATH, based on the suspension; filtering the ATH suspension thus forming a filter cake; optionally washing said filter cake one or more times with water.

5. The process according to claim 1 wherein said filter moist cake contains i) in the range of from about 55 to about 65 wt.% ATH particles; ii) in the range of from about 40 to about 70 wt.% ATH particles; iii) in the range of from about 40 to about 60 wt.% ATH particles; or iv) in the range of from about 45 to about 55 wt.% ATH particles; v) in the range of from about 25 to about 50 wt.% ATH particles; or vi) in the range of from about 30 to about 45 wt.% ATH particles; wherein all wt.% are based on the total weight of the filter moist cake.
6. The process according to claim 4 wherein said ATH product particles and/or the mill-dried ATH particles have an oil absorption, as determined by ISO 787-5:1980, in the range of from about 1 to about 35%, a BET specific surface area, as determined by DIN-66132, in the range of from about 1 to 15 m²/g, and a d₅₀ in the range of from about 0.5 to 2.5 μm.
7. The process according to claim 4 wherein the r₅₀ of the ATH product particles and/or the mill-dried ATH particles are in the range of from about 0.185 to about 0.325 μm and the V_{max} is in the range of from about 390 to about 480 mm³/g.
8. The process according to claim 4 wherein said ATH product particles and/or the mill-dried ATH particles have a d₅₀ in the range of from about 0.9 to about 2.2, a BET specific surface area in the range of about 3 to about 15 m²/g, an r₅₀ in the range of from about 0.185 to about 0.25 μm, a V_{max} in the range of from about 350 to about 550 mm³/g, and an oil absorption in the range of from about 19 to about 30%.
9. The process according to claim 2 wherein the oil absorption of said ATH product particles and/or the mill-dried ATH particles have an oil absorption in the range of from about 26 to about 32%.
10. The process according to claim 9 wherein the d₅₀ of said ATH product particles and/or the mill-dried ATH particles is in the range of from about 1.8 to about 2.2 μm.
11. The process according to claim 1 wherein the mill drying is effected by passing the filter moist cake through a mill drier operated under conditions including a throughput of a hot air stream greater than about 3000 Bm³/h, a rotor circumferential speed of greater than about 40 m/sec, wherein said hot air stream has a temperature of greater than about 150°C and a Reynolds number greater than about 3000.
12. The process according to claim 11 wherein the ATH particles in the filter cake have i) a BET in the range of from about 1.0 to about 4.0 m²/g and a d₅₀ in the range of from about 1.8 to about 3.5 μm; ii) a BET in the range of from about 4.0 to about 8.0 m²/g and a d₅₀

in the range of from about 1.5 to about 2.5 μm ; or iii) a BET in the range of from about 8.0 to about 14 m^2/g and a d_{50} in the range of from about 1.5 to about 2.0 μm .

13. The process according to any of claims 1, 2, or 11 wherein said process further includes b), and the number of any agglomerates present in said mill-dried ATH particles is reduced through the use of i) a dry mill; ii) an air classifier; or iii) any combination thereof.

14. The process according to claim 13 wherein the d_{50} of the ATH product particles is greater than or equal to 90% of the d_{50} of the mill-dried ATH particles.

15. The ATH product particles according to claim 1.

16. A process for producing ATH particles comprising:

- a) mill drying a filter moist cake to produce mill-dried ATH particles comprising agglomerates; and
- b) reducing the number of agglomerates present in said mill-dried ATH particles through the use of i) a pin mill; ii) air classifier, or iii) any combination thereof, thus producing ATH product particles,

wherein the filter moist cake contains in the range of from about 25 to about 85 wt.% ATH particles having a d_{50} in the range of from about 1.5 to 3.5 μm , and wherein said filter cake is obtained from a process that involves producing ATH particles through precipitation and filtration and the ATH product particles are characterized as having an oil absorption, as determined by ISO 787-5:1980, in the range of from about 1 to about 35%, an r_{50} in the range of from about 0.09 to about 0.33 μm , and

- i) a BET specific surface area of from about 3 to about 6 m^2/g ; and a V_{max} volume of from about 390 to about 480 mm^3/g ;

or

- ii) a BET specific surface area of from about 6 to about 9 m^2/g ; and a V_{max} of from about 400 to about 600 mm^3/g ;

or

- iii) a BET specific surface area of from about 9 to about 15 m^2/g ; and a V_{max} of from about 300 to about 700 mm^3/g .

17. The process according to claim 16 wherein said filter moist cake is obtained from a process that comprises dissolving aluminum hydroxide in caustic soda to form a sodium aluminate liquor; filtering the sodium aluminate solution to remove impurities; cooling and diluting the sodium aluminate liquor to an appropriate temperature and concentration;

adding ATH seed particles to the sodium aluminate solution; allowing ATH particles to precipitate from the solution thus forming an ATH suspension containing in the range of from about 80 to about 160 g/l ATH, based on the suspension; filtering the ATH suspension thus forming a filter cake; and optionally washing said filter cake one or more times with water.

18. The process according to claim 17 wherein said filter moist cake contains i) in the range of from about 25 to about 85 wt.% ATH particles; ii) in the range of from about 55 to about 65 wt.% ATH particles; iii) in the range of from about 40 to about 70 wt.% ATH particles iv) in the range of from about 40 to about 60 wt.% ATH particles; or v) in the range of from about 45 to about 55 wt.% ATH particles; vi) in the range of from about 25 to about 50 wt.% ATH particles; or vii) in the range of from about 30 to about 45 wt.% ATH particles; wherein all wt.% are based on the total weight of the filter moist cake.
19. The process according to claim 18 wherein said ATH product particles have a d_{50} in the range of from about 0.9 to about 2.2, a BET specific surface area in the range of about 3 to about 15 m^2/g , an r_{50} in the range of from about 0.185 to about 0.25 μm , a V_{max} in the range of from about 350 to about 550 mm^3/g , and an oil absorption in the range of from about 19 to about 30%.
20. The process according to claim 16 wherein the mill-drying is effected by passing the filter moist cake through a mill drier operated under conditions including a throughput of a hot air stream greater than about 3000 Bm^3/h to about 40,000 Bm^3/h , a rotor circumferential speed of greater than about 70 m/sec, wherein said hot air stream has a temperature of from about 150°C to about 450°C and a Reynolds number greater than about 3000.
21. The process according to claim 20 wherein said ATH product particles have an oil absorption in the range of from about 26 to about 35%.
22. The process according to claim 16 wherein the ATH particles in the filter moist cake have i) a BET in the range of from about 1.0 to about 4.0 m^2/g and a d_{50} in the range of from about 1.8 to about 3.5 μm ; ii) a BET in the range of from about 4.0 to about 8.0 m^2/g and a d_{50} in the range of from about 1.5 to about 2.5 μm ; or iii) a BET in the range of from about 8.0 to about 14 m^2/g and a d_{50} in the range of from about 1.5 to about 2.0 μm .
23. The process according to claim 22 wherein said ATH product particles have a d_{50} in the range of from about 1.8 to about 2.2, a BET specific surface area in the range of about 3 to about 6 m^2/g , an r_{50} in the range of from about 0.2 to about 0.3 μm , a V_{max} in the range of from about 410 to about 450 mm^3/g , and an oil absorption in the range of from about 25 to about 30%.

24. The process according to claim 22 wherein said ATH product particles have a d_{50} in the range of from about 1.4 to about 1.8, a BET specific surface area in the range of about 6 to about 9 m^2/g , an r_{50} in the range of from about 0.185 to about 0.25 μm , a V_{max} in the range of from about 450 to about 550 mm^3/g , and an oil absorption in the range of from about 26 to about 32%.
25. The process according to claim 22 wherein said ATH product particles have a d_{50} in the range of from about 0.9 to about 1.8, a BET specific surface area in the range of about 9 to about 15 m^2/g , an r_{50} in the range of from about 0.09 to about 0.21 μm , a V_{max} in the range of from about 350 to about 550 mm^3/g , and an oil absorption in the range of from about 27 to about 35%.
26. A process for producing ATH particles comprising:
- a) mill drying a filter moist cake in a mill drier operated under conditions including a throughput of a hot air stream greater than about 3000 Bm^3/h to about 40,000 Bm^3/h , a rotor circumferential speed of greater than about 70 m/sec , and said hot air stream has a temperature of from about 150°C to about 450°C and a Reynolds number greater than about 3000 thus producing mill-dried ATH particles comprising agglomerates, and
 - b) reducing the number of agglomerates present in said mill-dried ATH particles through the use of i) a pin mill; ii) air classifier, or iii) any combination thereof, thus producing ATH product particles;

wherein:

- i) the filter moist cake contains in the range of from about 25 to about 85 wt.% ATH particles having a d_{50} in the range of from about 1.5 to 3.5 μm ;
- ii) said filter moist cake is obtained from a process that involves producing ATH particles through precipitation and filtration; and
- iii) the dry-milled ATH particles are characterized as having an oil absorption in the range of from about 1 to about 35% and:
 - 1) a d_{50} in the range of from about 1.8 to about 2.2, a BET specific surface area in the range of about 3 to about 6 m^2/g , an r_{50} in the range of from about 0.2 to about 0.3 μm , a V_{max} in the range of from about 410 to about 450 mm^3/g , and an oil absorption in the range of from about 25 to about 30%;

or

- 2) a d_{50} in the range of from about 1.4 to about 1.8, a BET specific surface area in the range of about 6 to about 9 m²/g, an r_{50} in the range of from about 0.185 to about 0.25 μ m, a $V_{\max}$ in the range of from about 450 to about 550 mm³/g, and an oil absorption in the range of from about 26 to about 32%;

or

- 3) as a d_{50} in the range of from about 0.9 to about 1.8, a BET specific surface area in the range of about 9 to about 15 m²/g, an r_{50} in the range of from about 0.09 to about 0.21 μ m, a $V_{\max}$ in the range of from about 350 to about 550 mm³/g, and an oil absorption in the range of from about 27 to about 35%.

27. The process according to claim 27 wherein said filter moist cake is obtained from a process that comprises dissolving aluminum hydroxide in caustic soda to form a sodium aluminate liquor; filtering the sodium aluminate solution to remove impurities; cooling and diluting the sodium aluminate liquor to an appropriate temperature and concentration; adding ATH seed particles to the sodium aluminate solution; allowing ATH particles to precipitate from the solution thus forming an ATH suspension containing in the range of from about 80 to about 160 g/l ATH, based on the suspension; filtering the ATH suspension thus forming a filter cake; optionally washing said filter cake one or more times with water.
28. The process according to claim 28 wherein said filter moist cake contains i) in the range of from about 25 to about 85 wt.% ATH particles; ii) in the range of from about 55 to about 65 wt.% ATH particles; iii) in the range of from about 40 to about 70 wt.% ATH particles; iv) in the range of from about 40 to about 60 wt.% ATH particles; v) in the range of from about 45 to about 55 wt.% ATH particles; vi) in the range of from about 25 to about 50 wt.% ATH particles; or vii) in the range of from about 30 to about 45 wt.% ATH particles; wherein all wt.% are based on the total weight of the slurry or the filter moist cake.
29. The process according to claim 26 wherein the r_{50} of the ATH product particles is in the range of from about 0.09 to about 0.33 μ m.
30. A flame retarded polymer formulation comprising at least one synthetic resin and the ATH product particles according to claim 15.
31. A molded or extruded article made from the flame retarded polymer formulation according to claim 30.

32. A wire or cable coated with the flame retarded polymer formulation according to claim 30.

Figure 1

Specific pore volume as a function of the applied pressure for the second intrusion run and the inventive ATH grade in comparison with standard grades

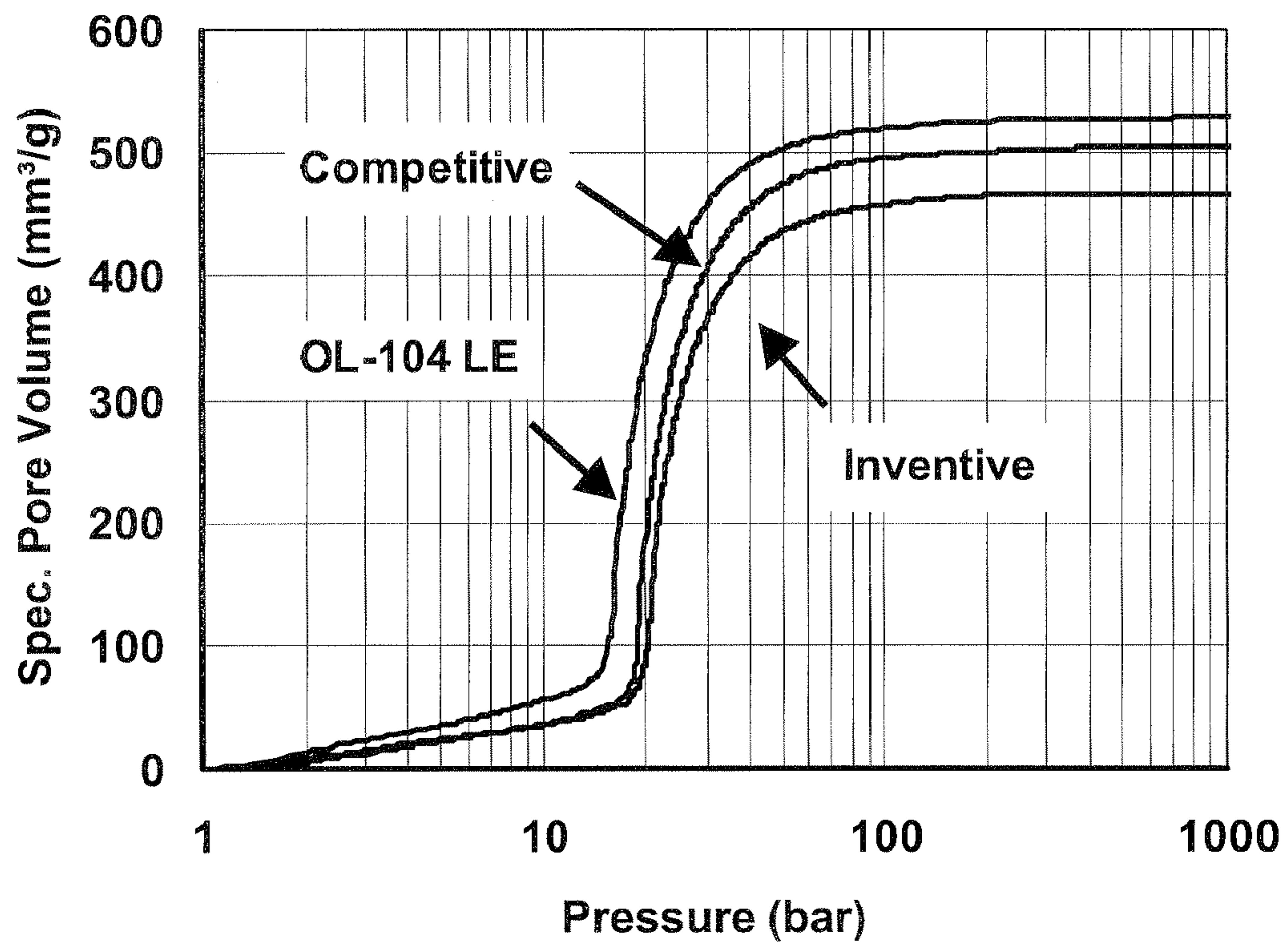


Figure 2
Specific pore volume as a function of the pore radius for the
second intrusion run and the inventive ATH grade in
comparison with standard grades

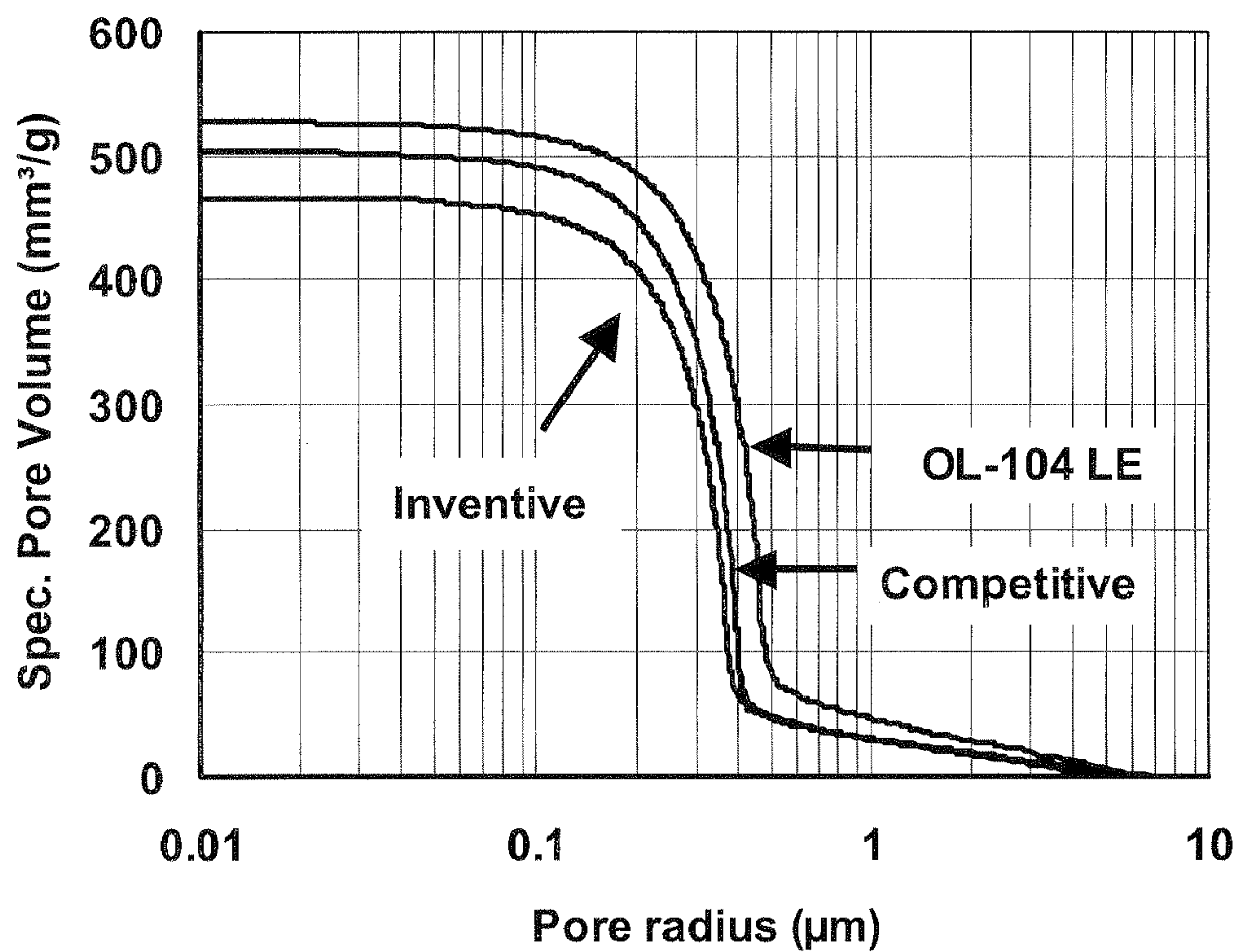


Figure 3

Normalized specific pore volume as a function of the pore radius
for the second intrusion run and the inventive ATH grade in
comparison with standard grades

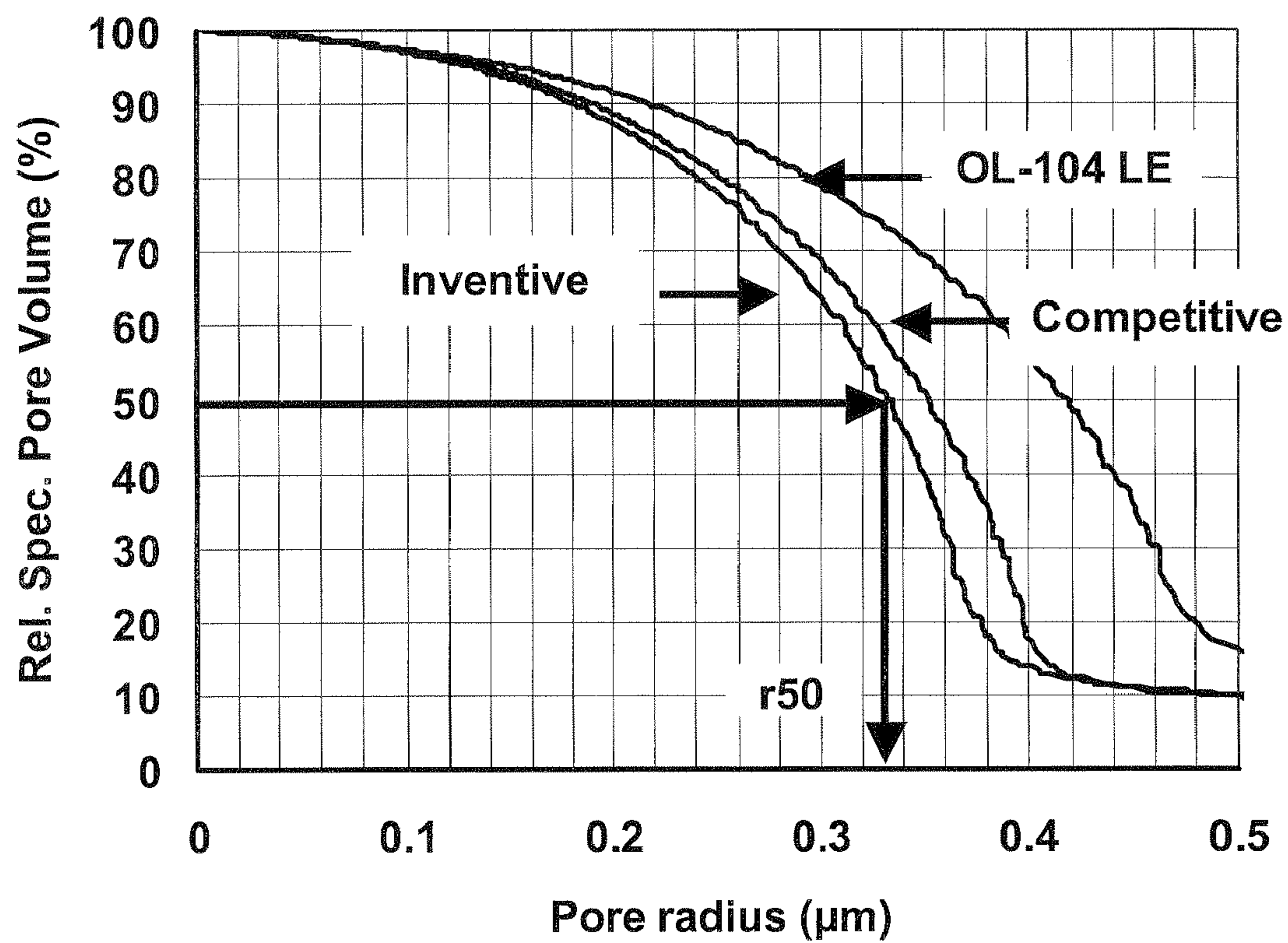


Figure 4

**Power draw on the motor of the discharge extruder of a Buss Ko-kneader
for the inventive ATH grade**

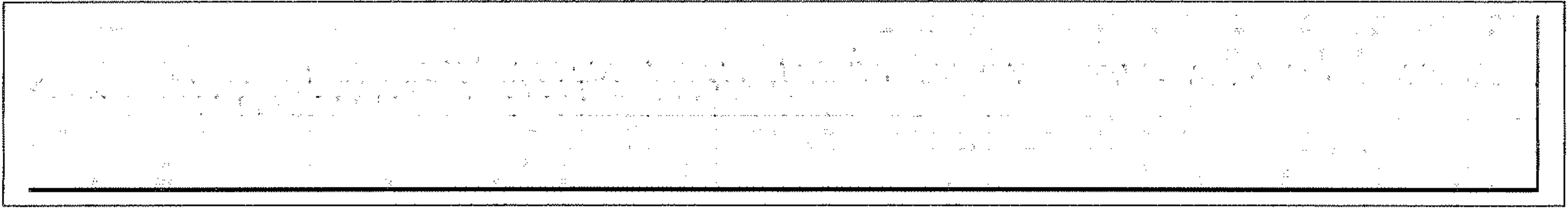
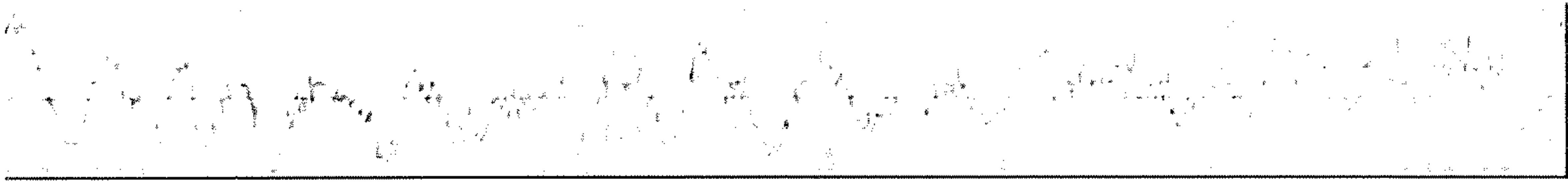


Figure 5

**Power draw on the motor of the discharge extruder of a Buss Ko-kneader
for the comparative ATH grade OL-104 LE**



Specific pore volume as a function of the applied pressure for the second intrusion run and the inventive ATH grade in comparison with standard grades

