Manufacture and Uses of Active Carbon

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The bulk of active carbon produced today is made from either coconut shells or coal-based materials, activated by selective oxidation with steam at elevated temperatures. This produces a product with the ability to effectively absorb even trace quantities of either unwanted or valuable liquids and gases. This is its main field of application, where it plays a very important part in solvent recovery processes, water and effluent treatment, and the treatment of flue gas before discharge to the atmosphere. These are all applications which are likely to expand in the future due to stricter anti-pollution legislation.

THE use of carbon dates back at least as far as the ancient Egyptians who used it for medicinal purposes. Its application expanded somewhat in 1773 when Scheele discovered the phenomenon of gas adsorption on charcoal, and 12 years later Lowitz successfully demonstrated its decolorisation capacity. This technique was first used by the sugar industry, which soon switched over from wood chars to bone chars when it was discovered that the latter were superior and could be regenerated many times.

Production of experimental carbons from a variety of basic materials followed the discovery of bone char, and liquid-phase operations continued to be the main area of application. Coconut shell as a raw material was first investigated about 1865 by Hunter, who reported its capacity for adsorbing vapour. Around the same time Lipsome produced a carbon for purifying potable water and Stenhouse suggested that carbon be used for ventilating sewers and also described the forerunner to the gas mask.

At the turn of the twentieth century manufacturing processes for active carbon began to appear along with a number of patents. But the event that stimulated the development of the high-quality carbons used today was the 1914–18 War, in which chlorine gas was used by the German army, and for which an effective countermeasure had to be found quickly. Since then the chemical industry has continued research into the production, characterisation and uses of active carbon over an ever expanding field of application.

Raw materials

Materials used in the manufacture of active carbons on an industrial scale include coconut shell, coals, wood and peat.

Coconut shell contains about 75% volatile matter and moisture, which is removed largely at the source by partial carbonisation in order to minimise shipping costs. The cellulosic structure of shell determines the end product which, at normal yields of 30–40% on the carbonised basis, is a material with a very high internal surface, consisting of pores and capillaries of molecular dimensions. The ash content

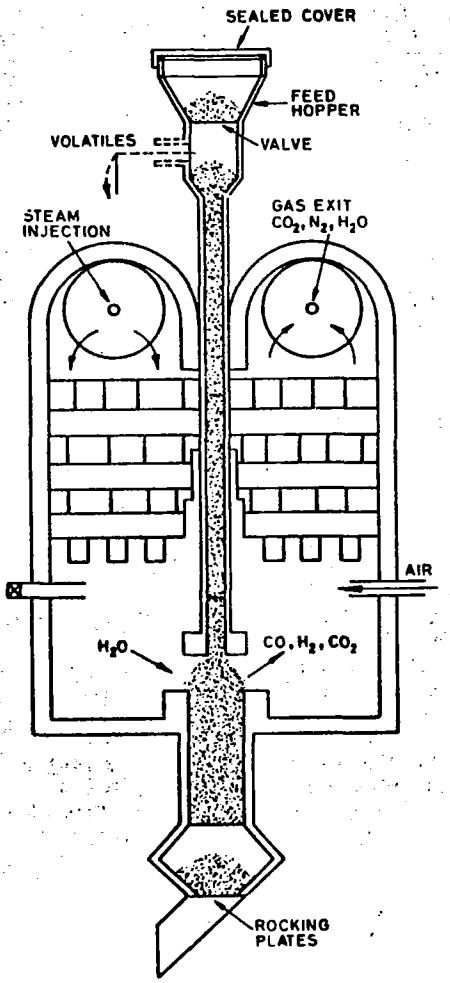


Fig. 1. Simplified diagram of an activated carbon retort

(Courtesy: Chemical Publishing, New York)

of such carbons is normally very low; the main ash constituents are dependent to some extent on the area from which the shells are obtained.

Coal is also a basic material which is, in parts of Europe and the US, readily available and reasonably cheap. The final product naturally depends upon the type of coal used and the initial processing prior to carbonisation and activation. It is normal procedure in some cases to grind the coal and reconstitute it into a form suitable for processing by the use of a binder such as pitch. Another method involves grinding the coals and utilising the volatile content as a binder by application of pressure. This second method allows for judicious blending of selected materials to control the swelling power of the coals and prevent coking and production of large pores which are unsuitable for adsorption purposes. Blending of coals also gives a degree of control over the structure and properties of the final product.

Wood is a useful raw material which can be activated by either one of two methods —steam activation or chemical activation, depending upon the product required. The better product is normally produced by chemical activation with phosphoric acid, which gives a char with a large surface consisting of pores of molecular dimensions suitable for adsorbing colour bodies from solution. The carbon is normally in a finely divided state and since it is produced from waste materials, such as sawdust, it is relatively cheap and can be used on a 'throw-away' basis.

Other techniques of chemical activation practised with wood and peat involve the use of zinc chloride and similar substances, the product being extruded, activated and subjected to various leaching processes to remove the zinc salt.[1,2]

Manufacturing processes

Great developments have occurred in the methods of manufacture of active carbons since early days when normal procedure involved the combustion of stacks of wood in inadequate air supplies. Material produced in this way has a very limited surface area and is completely uncompetitive when compared with modern products.

The bulk of present-day materials is produced from either coconut shells or coal-based materials, which are fed to a

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retort on a continuous basis. The normal activation process involves the use of steam at selected temperatures, selective oxidation of the material resulting in the production of carbons with pores of molecular dimensions. Shell carbon, being of a cellulose base, produces a material with a finer pore structure than is obtainable from coals, which require processing prior to activation if a viable product is to be obtained.

Retorts designed to produce active carbons (see Fig. 1) usually operate in one of three ways—vertically, horizontally or using a series of hearths, depending upon the method favoured by the particular manufacturer. In all cases, the quality of the product depends ultimately upon the residence time of the material in the 'activation zone'.

Activation in the vertical retort, utilising steam, is controlled by the rate at which material is withdrawn from the discharge hopper. The activation zone covers only a minor part of the total space available and it is possible, although not common practice when high quality is desirable, to proceed from raw to finished product in one pass.

After withdrawal from the retorts, the material is cooled and passed through a series of granulators and screens to give a carbon of a known quality which is available in a variety of grade sizes to suit most applications.

The alternative process for manufacture, involving the use of chemical digestion of sawdust, wood shavings or peat by phosphoric acid or zinc chloride, produces a material with much larger pores, since the process cannot be subjected to the same measure of control as steam activation.

The base materials normally contain large amounts of volatile matter and the advantage of chemical activation lies in the production of highly adsorptive materials at much higher yields than would be possible using steam.

Carbons produced in this manner, however, have one inherent failure—their lack of mechanical strength—and are not suited to vapour-phase operation, or to liquid-phase operation in a granular form.

Grades of carbon

A variety of grades and qualities of activated carbons are manufactured and these can be classifed as follows:

Coconut-shell-based carbons — for vapour-phase applications involving solvents of small molecular dimensions. They can also be used for a few liquid-phase operations.

Coal-based carbons—for catalyst-support applications, vapour-phase operation with medium- and relatively-high boiling solvents, and for liquid-phase operation.

Impregnated carbons—for mercury and radioactive iodine removal and for inclusion in cooker hoods to remove odour.

Powdered carbon—for decolouring, water treatment and medicinal purposes.

Gas/liquid recovery

Loss of reagent in a process directly affects the economics of the process and can also pose a pollution problem. Therefore it is essential to eliminate this loss, and here active carbon is an invaluable tool, particularly for recovering valuable solvents from processes—such as synthetic fibre and plastics manufacture—where evaporation of residual solvents is practised.

An active carbon system is normally designed to meet the specific conditions applying at any particular plant. Since conditions vary from place to place, it is not really possible to design a standard unit which can be made on a production line basis, unless a number of plants within a particular industry are to be served.

Concentration of solvent normally dealt with can vary from a few ppm to as much as 1·5–2% by volume in the air stream, according to the source. The effectiveness of solvent recovery depends a great deal upon its concentration and boiling point; the more dilute the concentration, the less efficient the process.

Other factors must also be considered, including the susceptibility of the solvent to reactions which can be catalysed by active carbon. These reactions could cause corrosion within the unit, or produce conditions conducive to ignition or explosion. These problems are common to certain classes of solvents which are recovered effectively by active carbon. They are overcome by careful selection of materials of construction and strict adherence to operating conditions specified by the manufacturers.

Other problems are posed by substances which carbon cannot readily adsorb. However, many of these can be solved by using the carbon as a base for a chemical substance which can either catalyse the decomposition of, or combine with, the gas in question. This application includes removing the contaminant from a radioactive iodine source and the removal of noxious substances such as hydrogen sulphide by oxidation to elemental sulphur.

Liquid effluents pose a different type of problem since the final product must either be suitable for reuse or, if intended for disposal, must attain standards set by local authorities or river boards.

Treatment may be carried out batchwise using powdered carbons, or, if possible, by the use of a percolation technique utilising granular carbon on a cyclic basis. Trace amounts of sparingly-soluble solvents are normally recovered in this way but the technique is not applicable to effluents containing solvents or other materials of a strongly polar nature.

Colour bodies, on the other hand, are generally composed of molecules with a relatively large cross-sectional area which can be adsorbed by carbon, provided that they are not strongly ionic. In general, systems used for this type of treatment incorporate powdered carbon containing a proportion of macropores on a 'throw-away' basis; it is also possible to use suitable grades of granular active carbon, regeneration being effected by high temperatures in presence of steam.

Water treatment

Many water supplies, such as river water, contain a proportion of sewage or industrial effluent and are unsuitable for reuse without further treatment. This treatment often involves dosage with powdered carbons in order to remove trace contaminants which give undesirable taste or odour characteristics to the water.

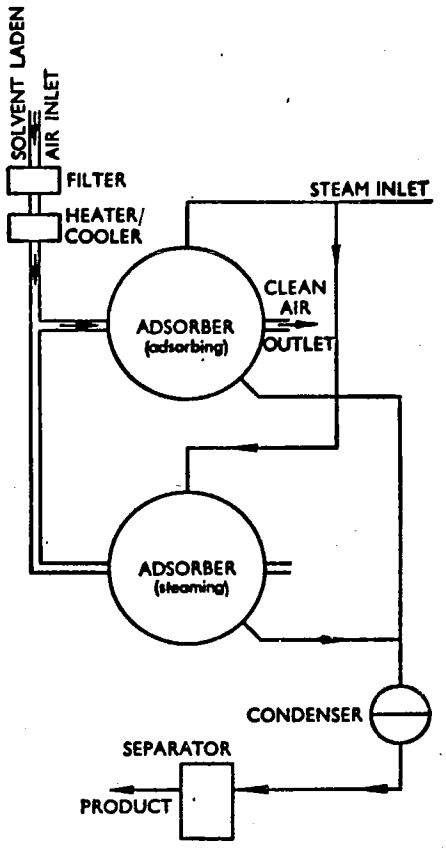


Fig. 2. Basic flow diagram of a gaseous-phase adsorption plant using active carbon for solvent recovery

Fig. 3. Flow diagram of the annular bed-type static solvent recovery plant

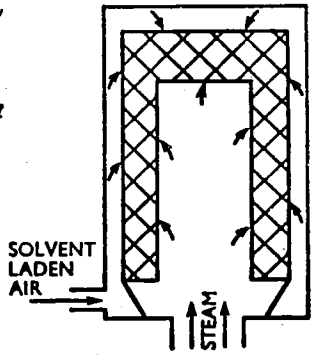
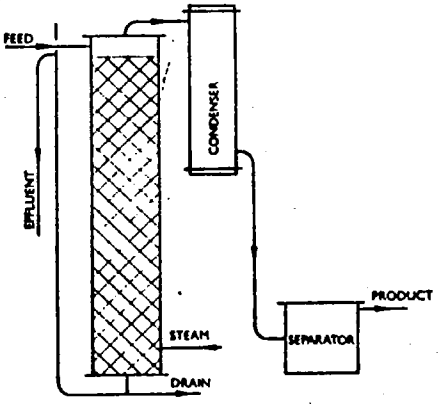


Fig. 4. Flow diagram of a liquid-phase adsorption unit



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Soft drink manufacturers and other industries requiring water of extra high purity, practise chlorination/dechlorination techniques to remove traces of organic colour and taste. Active carbon is used in granular form to remove traces of chlorine from the treated water. The reaction takes place on the adsorbent, being of a catalytic nature, and the life expectancy of the carbon is normally very high.

Attention is now being paid to reclamation of water, particularly in the US where extensive research programmes are being carried out into the treatment of industrial effluents, river water and even sewage effluent, as a means of combating the increasing shortage of suitable water supplies.

Basic research involving the use of active carbon is also being carried out now in the UK—on a much smaller scale than in the US—and results obtained so far are very encouraging and it is expected that greater interest will be generated as the need for new supplies increases.

It has been demonstrated by Slack[3] that the use of a coagulant, followed by active carbon in powder form, can produce an effluent from a sewage works of similar quality to the normal river intake of the district. The problem in this case is seasonal, however, and powdered material is used on a 'throw away' basis to minimise capital outlay. Further work is being carried out at Nottingham involving the purification of water from the River Trent using a granular carbon system.

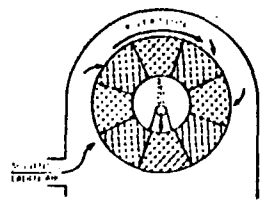
Treatment of this type results in the removal of organic material and contaminants, such as detergents, by adsorption on active carbon. The system can be operated on a cyclic basis, allowance being made for regeneration of the adsorbent by heat treatment at 800–900°C in the presence of steam.

Investigations are also proceeding into the treatment of raw sewage by physico-chemical methods involving precipitation and carbon treatment. Again, the investigation is in its early stages but effluents with a very low BOD (3·5 ppm) have been produced, and it is highly probable that such treatment will, in the near future, be considered as an alternative to traditional biological methods. Treatment of final effluent from an existing sewage works is quite feasible and reduces the detergent content to virtually nil whilst reducing the organic load, the net result being a higher grade effluent for disposal. This treatment could be of great significance in situations where one authority abstracts water from a point below the outfall of another authority's discharge.

It is apparent that active carbon has a big part to play in future developments and that it may well form the basis of an entirely new process resulting in the production of high-grade waters.

Solvent recovery processes

There are two main types of recovery unit: a static system involving the use of an annular carbon bed; and a continuous unit in which the carbon is packed into boxes which rotate around a horizontal axis. Small units utilising the flat-bed technique have also been manufactured occasionally to satisfy specialised needs.



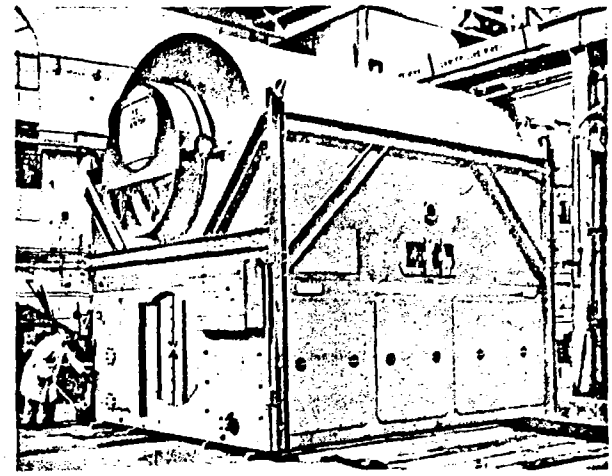
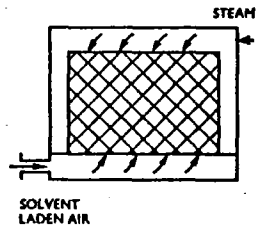


Fig. 5 (right). *Rotary bed plant for recovering 150 gal/hr of petroleum solvent from a 20,000 cfm air flow. A simplified diagram of the plant is shown above*



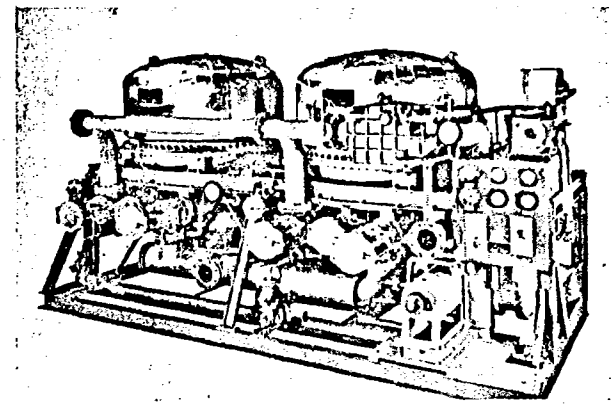


Fig. 6 (right). *Packaged-type, fully-automatic recovery plant of the cylindrical, flat-bed design. The flow diagram is shown above*

Solvent recovery operations from processes such as synthetic fibre spinning must be capable of dealing with a large air movement and solvent concentrations of the order of 1% v/v. It is usual, therefore, to design plant to suit the prevailing conditions and, due to the volume of air involved, a great deal of carbon is normally required to adsorb the available solvent.

Initial laboratory testing determines the capabilities of the adsorbent under the specified conditions and the ultimate number and size of adsorbers involved is determined by such characteristics as the volume of air available, velocity permissible and resistance to airflow of the adsorbent, which in turn determines maximum bed depth of the adsorbent.

The adsorbent is charged to a level which permits little or no solvent loss, a factor which can be regulated instrumentally; the solvent is stripped from the carbon by passing live steam through the bed in the reverse direction. The amount of solvent taken from the adsorbent is maintained at an economical level by controlling the steam input. Control of the complete system is normally carried out on a fixed time basis, desorption and drying periods being regulated by the number of adsorbers involved and the time required to charge the adsorbent to its working capacity. Basic flow diagrams for static-bed type of plant are illustrated in Figs. 2 and 3. Very high efficiencies are obtained using this system, as have been consistently demonstrated by the monitoring of effluent air from adsorbers on various sites, using a very sensitive hydrocarbon analyser.

Ancillary equipment is dependent upon the solvent or solvent mixtures involved and includes distillation stages to effect separation. Or, in some cases, an aqueous phase containing a low concentration of a sparingly-soluble solvent may be stripped using a percolation technique, the basis of which is demonstrated in Fig. 4.

The continuous unit is a much more compact plant, the carbon being packed into a series of boxes which form segments of a horizontal rotor. Each box traverses a full cycle which consists of adsorption, desorption and drying stages, a complete cycle representing one complete rotation. The efficiency of the carbon is determined experimentally and the speed of rotation is calculated from a knowledge of the available solvent and the capabilities of the adsorbent.

This type of unit is suited to applications such as the recovery of hydrocarbon solvents in the printing industry and is popular in the US, where there is a demand for package-type units. An illustration is given in Fig. 5. Another package-type unit of flat-bed design, manufactured for specialised conditions and utilising titanium to resist severe corrosion, is illustrated in Fig. 6. Typical performance data which can be expected from Sutcliffe, Speakman carbons, operating with a variety of solvents, are given in Table 1.

Future developments

Pollution of the environment is one of the big issues of today and active carbon will have an ever increasing part to play in the control, reduction or elimination of conditions responsible.

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Table 1. Some typical results of solvent adsorption by carbons, as experienced in solvent recovery practice

Solvent	Concentration (% w/w)	*Initial adsorption (% w/w)	†Cyclic adsorption (% w/w)	‡Steam ratio	Boiling point range (°C)
Methylene chloride	1·0	28·3	17·3	1·4	40·5
Arklone P	0·5	44·9	20·8	1·4	47·6
Acetone	1·0	20·3	12·5	2·3	56
Tetrahydrofuran	0·5	22·0	9·0	2·0	66
Hexane	0·48	21·3	8·2	3·5	68·7
Ethyl acetate	0·5	27·6	13·6	2·1	77·2
Trichlorethylene	0·5	44·6	19·9	1·8	86·7
n-Heptane	0·12	22·4	5·9	4·3	98
Toluene	0·4	23·3	9·6	3·5	110
Methyl isobutyl ketone	0·2	22·0	9·0	3·5	115·9
Mixed rubber solvent	0·3	25·7	10·3	3·8	120–160
Shellsol E	0·3	35·6	11·9	3·7	153–193
3,3,5-trimethyl cyclohexyl acetate	0·1	36·4	7·9	4·5	206

*Initial adsorption refers to the solvent adsorbed by new carbon to 'breakpoint', i.e. when solvent can be detected in the effluent air stream. The 'slip' level at this point is normally of the order of 20 ppm.

†Cyclic adsorption refers to the solvent which can be adsorbed and desorbed consistently at an economical steam consumption.

‡Steam ratio refers to the total steam used to desorb the solvent from the adsorbent and is an overall figure inclusive of condensate and moisture condensed on the carbon bed.

The use of carbon in water treatment is the subject of much research at present and it is likely that, within the next few years, new processes will be developed to produce reusable and even potable water from contaminated industrial and sewage effluents.

Air treatment will also increase in importance and future trends may dictate that solvent recovery operations will become necessary to remove trace contaminants from effluent air systems in order to comply with legislation. Treatment of flue gases to reduce oxides of sulphur and other contaminants—which are at present vented to atmosphere to become someone else's problem—may become the subject of clean air legislation. In this case active carbon may also have an important part to play in adsorbing the sulphur oxides which could then be recovered as elemental sulphur or sulphuric acid.

Active carbon is also being used as a filter, in applications such as the filter hoods for removing cooking odours and in some cigarettes, where active carbon in powdered or granular form is incorporated in the cigarette filter.

Reducing atmospheric pollution by motor vehicles is also receiving a great deal of attention and a device has been developed which removes petrol vapours by adsorption with carbon. The adsorbed material is then desorbed from the carbon by hot gas when the engine is running and returned to the engine. Legislation is already in force in certain US states whereby all new motor vehicles must be fitted with this device, and the demand for suitable carbon will increase as the laws are extended.

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process control system

The *Protronic* process control system of Hartmann & Braun AG—introduced last year at the ACHEMA Exhibition—represents a comprehensive automatic measuring and control system specially designed for the chemical and petrochemical industries where hazardous areas of operation exist. The circuits and instruments have been designed with special emphasis on explosion protection, signal interconnection techniques, computer compatibility, assembly and wiring.

The system uses intrinsically-safe two-wire transmitters for absolute and differential pressure, temperature and angle of rotation. To fully utilise the two-wire technique, a number of transmitter supply units have been designed together with safety barriers which allow transition from intrinsically-safe to non-intrinsically-safe circuits. The transmitters are not fed with a supply voltage; instead they function with a 'live zero' receiving power from a continuously-flowing basic current of 4 mA. The signal current is superimposed on to this up to a value of 20 mA. The transmitter supply unit has a supply voltage limited to 20 V by Zener diodes, and, together with the safety barriers, conforms to all VDE regulations on intrinsically-safe circuits. The low voltage of the supply unit allows a greater distance between the transmitters and the control room—an important point on very large plants.

In the control room the panel instruments —comprising controllers, ratio, set-point and manual stations, indicators, and recorders with selection stations—are all of modular design with identical front dimensions, case depths and standardised connections and terminal lineup. There are two types of process controller: the *B* model with a band scale and control deviation and the *F* series with a fixed scale and controlled variable indication. Within the two types there are various possible configurations of controller. The simplest *E* model has all functional groups mounted permanently while in the *S* model the input circuits and control amplifiers are designed as plug-in components. By replacing these components a change in the controller function and in the setting ranges of the parameters can be accomplished easily. Also additional plug-in units can be inserted such as output limiters, start-up circuits, etc.

There are also controllers compatible with process computers, such as controller *BM*. This is similar to the type *BS* controller except that, in addition, it features motor-driven set-point setting, remote-control logic and a memory amplifier for supervisory control with a process computer. If direct digital control is employed an analogue standby controller is often required in case the computer breaks down, and this is provided in the *Protronic* system by controller *FS*.

Since its introduction, Hartmann & Braun's new control system has been accepted widely by the German chemical industry and a large number of systems have been successfully installed there. The company are now planning to market the system in other countries through their subsidiaries in the UK, Holland, France, Italy, Austria, Australia, the US, and Brazil.

slurry consultants

Pierson & Co. (Manchester) Ltd. have established a special department dealing with slurry processing consultancy as an extension to their existing activities in this field. Through their many international associates, Pierson have been able to build up a network of contacts, all dealing with different facets of slurry technology. The consultancy service has already had some success particularly in effluent processing. Through their associate companies Pierson are able to offer a complete package deal as far as actual equipment is concerned, ensuring that any advice given is guaranteed 100%. The consultancy work can be undertaken on an overall-fee or daily-rate basis.

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